JANUARY, 1953
IN THREE SECTIONS—SECTION I

Journal of METALS

January JOURNAL OF METALS Highlights:

"Chuquicamata—A Story of Chilean Copper" - p. 19
"Rammed and Castable Refractories" - p. 34
"Select Engineer Employees Scientifically" - p. 37

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Journal of METALS

VOL. 5, NO. 1 — JANUARY, 1953

IN THREE SECTIONS — SECTION 1

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COVER

Copper matte is poured to converter in Chuquicamata's smelter, as years of planning and months of construction culminate in first copper from the new sulphide plant. See story starting on page 19.

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This issue of Journal of Metals is in three Sections—
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MEET THE AUTHORS

N. I. ANANTHANARAYANAN

R. J. SLADEK

N. I. Ananthanarayanan (p. 79), born in Crankandre, India is presently working towards his Ph.D. at Lehigh University. He had been employed as a research physicist and engineer in Bombay from 1944 to 1946. He enjoys chess, public speaking, and listening to Indian musical instruments.

R. J. Sladek (p. 69) is at present an AEC predoctoral fellow in physics at the University of Chicago, Institute for Study of Metals. Born in Chicago, Dr. Sladek is a graduate of the University of Chicago. From 1949 to 1951 he was a research technician at the University. A member of the American Physical Society, he counts reading as his major hobby.

H. A. DOERNER

R. F. DOMAGALA

H. A. Doerner (p. 39) retired from the Bureau of Mines in July 1952 and is now metallurgical consultant in San Francisco. A native of Denver, Mr. Doerner graduated from the University of Colorado in 1913. After graduation he joined the Western Chemical Co. and then accepted an appointment at the Colorado Agricultural College teaching chemistry. A year later Mr. Doerner joined the Bureau of Mines in Denver as a junior chemist in radioactivity. From 1916 to 1922 he had several jobs in private industry and became proficient in the handling, concentration, and refining of radium salts. He returned to the Bureau of Mines at Reno and was later transferred to Berkeley. He remained there for five years and then took charge of a research program in cooperation with the Mining Experiment Station at Washington State College, Pullman, Wash. Mr. Doerner was named chief of the nonferrous metals branch at Albany, Ore. and then became chief of the metallurgical div., Region III, San Francisco. He has presented several papers before AIME and enjoys contract bridge as his hobby.

R. F. Domagala (p. 73) is a graduate of the Illinois Institute of Technology. A member of the Chicago Section, AIME, Mr. Domagala has been connected with the Armour Research Foundation since graduation from college. He is now assistant metallurgist, physical metallurgy branch and his duties include phase diagram determinations. Tennis and golf occupy him in his free time.

P. D. S. ST. PIERRE

L. GUTTMAN

P. D. S. St. Pierre (p. 41), born in England and a graduate of the Royal School of Mines, London, in 1945, is presently residing in Ottawa. Dr. St. Pierre is a metallurgist in the mineral dressing and process metallurgy div., Dept. of Mines and Technical Surveys. In 1947 he was visiting mines and smelters in the United States and Canada. Ceramics are the hobby of this author.

Lester Guttman (p. 81) is a graduate of University of Minnesota (B.Chem. 1940) and University of California (Ph.D. 1943). Dr. Guttman was at Los Alamos for three years and in 1946 joined the staff at the University of Chicago, where he is now assistant professor. Dr. Guttman enjoys sailing and madrigal singing as his hobbies. He previously has authored papers for AIME.

J. H. KEELER

E. J. RIPLING

J. H. Keeler (p. 44), a research associate at the General Electric research laboratory at Schenectady, was born in Toledo. A graduate of Pennsylvania State College (B.S., 1942 and Ph.D., 1951) and Stevens Institute of Technology (M.S., 1948), Dr. Keeler had been associated with the Wright Aeronautical Corp., Wood Ridge, N. J. from 1943 to 1947. He then became a research assistant at the Pennsylvania State College. As his hobbies this AIME Member enjoys music, natural history, and stamp collecting.

E. J. Ripling (p. 66), born in Lewistown, Pa., has been senior research associate and assistant professor at Case Institute of Technology since 1946. Dr. Ripling graduated from Case and Pennsylvania State College. For one year he was a metallurgist with the Westinghouse Electric Co. In 1943 he joined the Copperweld Steel Co. and in 1944 was in the Navy. Following his discharge, Dr. Ripling joined the staff at Case. He has coauthored several papers and was one of the authors of the book *Tube Producing Practice*.

H. A. Johansen (p. 63), chemical engineer with the U. S. Bureau of Mines, Northwest Electrodevelopment Laboratory, is a native of Portland. He attended Reed College (1948) and University of Oregon (1950). Now residing in Albany, Ore., gardening and mountaineering are his hobbies.

J. BURBANK

H. L. GILBERT

Jeanne Burbank (p. 55) was born in Philadelphia. She is a graduate of the American University and University of Pennsylvania. From 1940 to 1946 Mrs. Burbank was associated with Peacock Laboratories, Inc. She then joined the Naval Research Laboratory, where she is presently physical chemist in the electrochemistry branch. A member of the American Chemical Society, Electrochemical Society, and AAAS, Mrs. Burbank enjoys ballet, art, and music. Her 10 year old daughter also manages to keep her busy.

H. L. Gilbert (p. 63) joined the U. S. Bureau of Mines after serving as an officer in the Chemical Warfare Service from 1942 to 1946. Born in Corvallis, Ore., he is a graduate of Oregon State College (B.S., 1942). Since his association with the Bureau, Mr. Gilbert has been engaged in chromium and zirconium research, development of Kroll process equipment, high vacuum melting, and consumable electrode arc melting techniques. He is head of the research and development section, rare metals branch at the Northwest Development Laboratory, Albany, Ore. He has co-authored AIME papers and his hobby is gardening—"no quantity, no quality, just size."

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Box A-2 JOURNAL OF METALS

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Box A-1 JOURNAL OF METALS

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METALLURGIST: Thoroughly conversant with Tool Steels. Should be able to handle customer complaints and write reports. Should also be able to conduct research under guidance. Salary dependent upon experience and initiative. Address replies: Firth Sterling Inc., McKeesport, Pa.

METALLURGIST or CHEMICAL ENGINEER. A graduate between the ages of 25 and 32 will find the work challenging and a step toward further advancement. The position vacant is that of plant metallurgist at the Dumas, Texas, zinc smelter. The duties consist of 50% office work, including the preparation of the monthly metallurgical report, and 50% process trouble shooting in the plant. The starting salary would be made commensurate with the ability and experience of the applicant. Write directly to Assistant Chief Metallurgist, American Zinc Co. of Illinois, P. O. Box 495, East St. Louis, Illinois.

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Henry Krumb Scholarships
William Campbell Fellowships

Applications are invited for the Krumb Scholarships and Campbell Fellowships for the academic year 1953-54. These are available to candidates for degrees in the fields of Mining, Mineral, and Metallurgical Engineering.

The Krumb Scholarships, with yearly stipends of $1000 and transportation to New York City, are in support of studies toward the B.S., M.S., or professional engineering degrees. Preference is given to students in mining engineering in the ratio of three in this field to two in metallurgical and mining engineering.

The Campbell Fellowships, established to encourage research in the general field of metals, are available to candidates for graduate and professional degrees including the Ph.D. Five grants are usually awarded each year, with stipends of $1800 for single students and $2100 for married students. Application blanks and further information may be obtained on request to the Office of University Admissions, Columbia University, New York 27, N. Y. Complete applications should be returned before Feb. 20, 1953.

— Personnel Service —

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service, Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., **New York 18;** 100 Farnsworth Ave., **Detroit;** 57 Post St., **San Francisco;** 84 E. Randolph St., **Chicago 1.** Applicants should address all mail to the proper key numbers in care of the New York office and include 6c in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service, to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for $3.50 a quarter, $12 a year.

POSITIONS OPEN

Engineers. (a) Metallurgical engineer capable of supervising a metallurgical laboratory both with respect to experimental work and to applied research on production problems. Preferably with an advanced degree or at least considerable experience in metallurgical techniques. (b) Metallurgists, young, for staffing the laboratory and carrying out research projects and investigations under the direction of a chief metallurgist. Salaries open. Location, Pennsylvania. Y7937.

Engineers. (a) Assistant chief engineer, 35 to 40, experienced in medium to heavy metal processing. Should know tooling and design for production. Should be a good administrator, as applicant will eventually head department. Salary open. (b) Sheet metal engineer, 35 to 40, with considerable experience in the application of sheet metal such as is used in refrigerators, cabinets, deep freezers, etc. Will also eventually head department. Salary open. Location, Ohio. Y7914.

Chemical or Metallurgical Engineer, under 30, for liaison duties between laboratory of company producing fuel pumps, carburetors, and other automotive components and customers. Should be able to meet and deal with people, analyze trouble and make logical recommendations regarding product improvement. Salary, $4800 a year. Location, East. Y7890-D8139.

Tool and Die Designer, 35 to 45, with at least five years' experience in die design of light metal stamping

WANTED

Metallurgist with experience or training in nonferrous metallurgy. Powder metallurgy experience desirable but not essential. Work on research and development of special products in small community in northwestern Pennsylvania.

Box A-3 JOURNAL OF METALS

and forming variety. Knowledge of progressive dies. Will design stamping and forming dies for light metal manufacturing. Will understudy chief engineer who retires in about three years. Some customer contact for a manufacturer of metal products. Salary, $6000 to $10,000 a year. Company will negotiate fee. Location, Illinois. Y7863-T9348.

Stress Analyst, 30 to 40, with at least five years' experience in wirecraft or other industry, including considerable stainless steel and high temperature alloys for metal products equipment manufacturer. Must be citizen with Q clearance. Salary, $6000 to $7200 a year. Location, New York, N. Y. Y78424.

Metallurgical Chemist, chemical or metallurgical graduate, with 2 to 3 years' experience for corrosion research on nonferrous alloys. Salary, $4800 a year. Location, northern New Jersey. Y7569.

Powder Metallurgist with a few years' practical experience for the development of powder metal products of many varieties of metals and shapes and the design of the plant and equipment to make them in quantity. Salary open. Location, western Pennsylvania. Y7561.

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Research Engineers, physical and inorganic chemists, chemical engineers and metallurgists to conduct research in any of the above fields. Location, Ohio. Y7621.

Foundry Superintendent, 35 to 55, to direct production of aluminum foundry. Must have at least ten years' experience in nonferrous foundry supervision, and knowledge of aluminum sand casting work. Salary, $7200 to $8400 a year. Location, New Jersey. R9384.

Metallurgical Engineer for large mining organization, to conduct test work preliminary to operation of a large copper-zinc reduction plant. Broad experience essential in roasting, leaching and zinc electrolysis. Salary open. Location, eastern Canada. Y7971.

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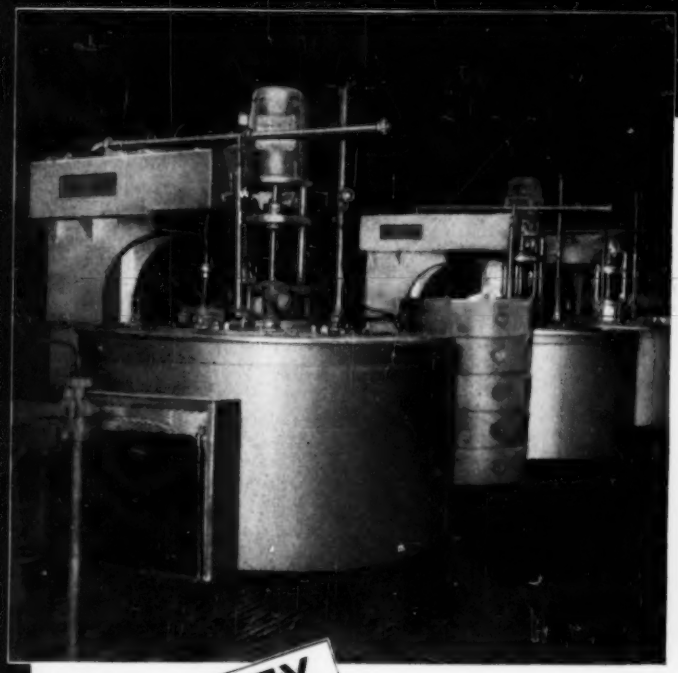

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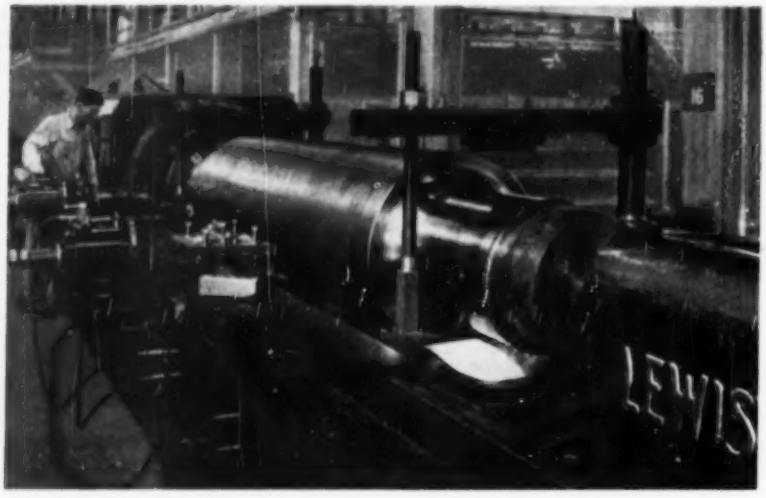
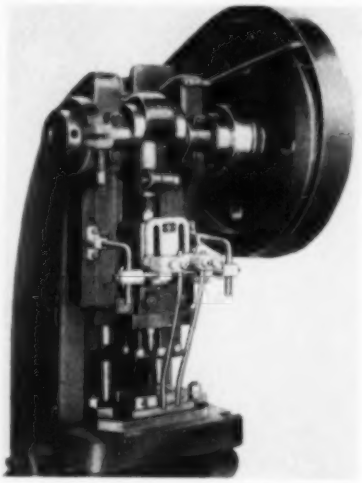
3—COLD CLEANERS: Used at room temperature in power washers these new cold cleaners developed by *E. F. Houghton & Co.* effectively remove sulphurized cutting oils, pigmented drawing compounds, and rust preventives from ferrous metals, copper, and brass. No heat is required and there is no steam or toxic mist.

4—LABORATORY STIRRER: This 1550 rpm laboratory stirrer features the *Eberbach Corp.* hollow spindle, which enables the user to raise or lower the propeller quickly. The degree of agitation can be varied by changing style of the propeller. It develops a maximum torque of 5.195 in.-oz.

5—HYDRAULIC ROLL LATHE: This machine is equipped with a 60-hp main drive motor with variable speed from 300 to 1200 rpm, and a separate motor for the hydraulic pumps. The pumps are designed to maintain a hydraulic pressure of 1000 lb in each of the cylinders. The newly developed lathe permits machining of very hard rolls which formerly could be surface-conditioned only by grinding. *Blaw-Knox Co., Lewis Foundry & Machine Div.*



6—SWEEP GUARD: A safety guard for forming and secondary operations on power presses up to 4 in. stroke is available from *Copper Weymouth, Inc.* Actuated by the press ram, two guard arms push the operator's hand away with a sweeping motion from the danger area as the ram starts to descend. Field of vision is unobstructed and the guard arms may be arranged in a variety of ways to suit specific applications.



7—FURNACE: *Detroit Electric Furnace Div., Kuhlman Electric Co.* has introduced a new investment casting furnace. It consists of a melting chamber and transformer combined into a single unit. The operator pours the heat by inverting the furnace and allowing the molten metal to run into the clamped-on mold. As soon as the casting has solidified, he releases the air pressure and removes the mold before returning the furnace to the melting position.

8—MATERIALS HANDLING: A hydraulically elevated truck unloader that can unload 20,000 lb of coke from a trailer truck in 1 min has been built by *Kewanee Mfg. Co.* The unloader tilts both the tractor and trailer to a 40° angle, dumping the coke into storage facilities.

9—BALL MILLS: Tungsten carbide lined ball mills are available from the *American Electro Metal Corp.* for the manufacture of fine powders, particularly of metals, ceramics, and other hard materials. Because of the inherent resistance of tungsten carbide to abrasion, contamination of the powders being milled by the ball mill is greatly reduced and often completely avoided. The ball mills are available in sizes up to 12 in. ID and 15 in. high.

10—FLOW METER: Low differential flow meter for measuring air flow, gas flow or recording the ratio of air flow to gas flow is announced by the *Hays Corp.* Since it uses a dry diaphragm type measuring element no water, oil, or mercury are required and leveling is eliminated.

11—PROTECTIVE COATINGS: Coatings specially formulated to protect equipment against corrosive conditions have been announced by the *Ric-Wil Plastic Coating & Mfg. Corp.* Ricwilite 1060 phenolic resin coating is of the baking or heat hardening type. Ricwilite 7100 phenolic resin coating is the cold setting type which is cured or polymerized at room temperatures, 60 to 80°F.

12—VIBRA-TAK: A pocket-size instrument for accurately measuring the speed and amplitude of vibration on machinery or on bins and hoppers where vibration is produced intentionally to aid materials movement, is now available from the *Martin Engineering Co.* It has a movable tuning slide that indicates on a number scale the speed of vibration.

13—TORQUE BALANCES: A line of precision torque balances manufactured in Holland has been intro-

duced in the United States by *Ohaus Scale Corp.* In drawing and finishing of tungsten wire or similar materials it becomes impractical to measure in any other manner than weight per unit length. A special wire cutting device for tungsten or other wire is available as an accessory.

14—GATE, RISER REMOVAL: A new radial type gate and riser cut-off machine for all types of nonferrous castings said to perform in one simple operation complete gate and riser removal, is announced by the *Acme Saw & Mfg. Co.* The cutter carriage moves laterally on a 6-ft swinging arm which is counter balanced with a 15 hp motor on the opposite side of the 10 in. supporting column.

15—SCREW CONVEYOR: *M-H Standard Co.* carries 6 in. diam stainless screw conveyor for immediate delivery. Standard sections are right hand 6 in. diam, 6 in. pitch x 9-10 in. long, ⅛ in. thick type 304 stainless steel, flight welded to 2 in. stainless steel pipe which is bushed and drilled for standard 1½ in. diam

coupling shafts. The entire screw is polished to a No. 4 finish. Left hand and all standard and special sizes from 4 to 12 in. are available.

16—PHOTOELECTRIC COUNTER: A packaged, easily installed, general purpose photoelectric counter is available from *Photoswitch, Inc.* Speed of operation is up to 600 counts per min.

17—MARKING INK: A fast drying marking ink that will not chip or peel and is unaffected by heat and weathering is offered by *John P. Nissen, Jr., Co.* It is ideal for marking rough castings, bar stock, tools, dies, etc.

Free Literature

20—INTERFERENCE FILTERS: A complete selection of transmission type interference filters for use in isolating specific spectral bands has been announced by *Bausch & Lomb Optical Co.* A descriptive brochure is available on how to get the most out of them, giving characteristics of the new films and suggestions.

21—MICROFILM: A service for placing records permanently on microfilm is outlined in a booklet published by *Remington Rand.*

22—SPECTROGRAPH CHART: Chart showing characteristic secondary X-ray beams for elements from sodium (atomic No. 11) to uranium (atomic No. 92) is available from *North American Philips Co., Inc.* It also shows K Alpha and K Beta lines for mica (reflecting) analyzing crystal.

23—AIR COMPRESSORS: Oil-free air compressors manufactured by *Joy Mfg. Co.* are discussed in bulletin A-92. Specifications shown include the complete line of nonlubricated compressors from 172 to 8800 cfm, 25 to 600 hp.

24—TWIST DRILLS: Catalog of twist drills has been published by the *DoAll Co.* Straight and taper shank for both general and special use, heavy duty and high helix drills, and three and four fluted drills in both straight and taper shank styles are covered.

25—MACHINED PARTS: Folder describing equipment and facilities for the production of machines parts has been issued by *Stryker Machine Products Co.* Machines available and a complete list of equipment are given.

26—SEAMLESS TUBING: Background information on *Superior Tube Co.'s* complete line of tubing is given in bulletin 32. An increase in the size range of seamless lightwall tubing from 1¼ in. OD max to 2 1/16 in. OD max has been announced.

27—STEEL SELECTION: Characteristics, mechanical properties, and uses of hot rolled and cold finished carbon and alloy steel bars are covered in condensed form in bulletin available from *Joseph T. Ryerson & Son, Inc.*

28—TUBE FURNACE: The new *Lindberg Engineering Co.* gas fired vertical radiant tube furnace is described in bulletin. A listing of design and application features and diagrams of this radically new furnace are contained in the bulletin. The furnace may be used for carbonitriding as well as carburizing, annealing, carbon restoration and other hardening jobs.

29—HOOK-ON INSTRUMENTS: *General Electric's* complete line of hook-on instruments is described in booklet. Applications, operation, and basic features of the type Ak-1A volt-ammeter; Ak-2 wattmeter; and Ak-3 power factor meter are included.

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January

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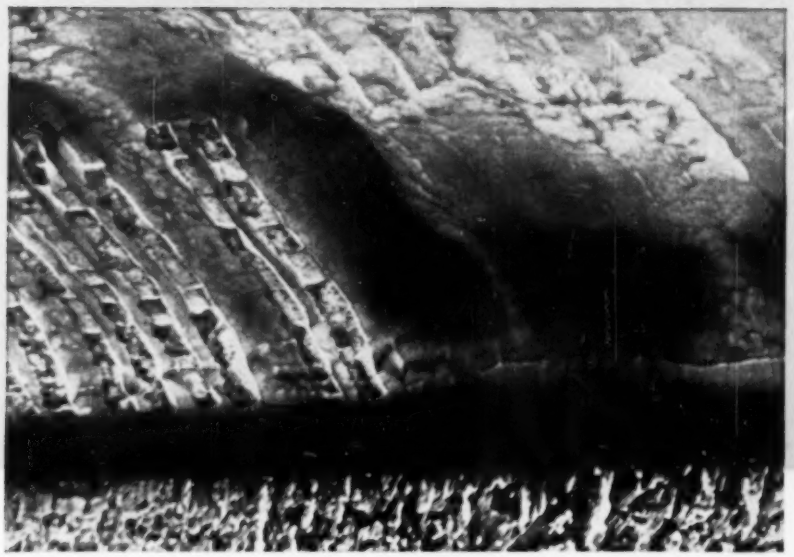
Survival of the fittest is the rule of the jungle. And so it is with refractories in the critical areas of the nation's open-hearth furnaces.

Convincing proof that Zebra roofs with Grefco's STEELKLAD-40 are tops in zones of greatest wear, has caused steel men to select STEELKLAD-40 in more than 9 out of 10 of existing Zebra roofs. Results often show more heats per campaign, reduced repairs—hence greater furnace availability.

STEELKLAD-40 is a patented unburned chrome-magnesite roof brick that is permanently jacketed in a steel shell which becomes an integral part of the brick itself. Developed in Grefco's modern research laboratories, STEELKLAD-40 is the major reason for the amazing increase in popularity of the open-hearth Zebra roof.

In service the STEELKLAD-40 key brick protrude beyond and protect the silica brick from destructive gases. The silica brick



make possible a strong, mechanically stable roof. Note the illustration above. The dark area to the right consists of plain silica construction. The silica brick are ready for a patch. Yet the Zebra section is still in good condition.

Surveys have proved that the Zebra roof has gained industry popularity faster than any other construction in open-hearth history. More and more steel men are calling on Grefco to help solve roof problems with STEELKLAD-40.

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Books for Engineers

Principles and Methods of Sheet-Metal Fabricating, by George Sachs. *Reinhold Publishing Corp.* $11.00, 526 pp., 1951.—Claims are made for the book as the outstanding reference work in its field. Numerous sheet metal forming methods are described systematically and in great detail. Similarities and differences are emphasized. The theory of deformation of sheet metal and tubing; the metallurgical effects of stamping, drawing, extruding, and bending; the practical operations involved, the design of parts and dies; and production equipment used, are all treated in the book.

Hydrometallurgy of Base Metals, by George D. Van Arsdale, prepared with the assistance of the Dorr Co. staff. *McGraw-Hill Book Co.* $9.50, 370 pp., 1953.—The recovery of base metals by leaching is fully and practically covered. With special reference to copper, the book gives much helpful technical and economic information on the hydrometallurgy of lead, zinc, antimony, cadmium, vanadium, uranium, and radium. Included is information on uranium leaching as recently developed in the Colorado Plateau area, the treatment of cobalt in Rhodesia, production of electrolytic manganese and antimony, and the heap leaching of copper ores. Processes and plants, equipment, materials, and economic considerations are detailed. The last two chapters cover the history and future of hydrometallurgy.

The Statistical Year Book, 1952—Tin; Tinplate; Canning. *The International Tin Study Group.* $5.60, 268 pp., 1952.—The book is divided into two parts. The first section contains a series of articles covering the events of recent years in the tin and tinplate industries. The second part contains a statistical account of the tin, tinplate, and canning situation in 110 countries. General articles describe attempts to the Tin Study Group and others to reach some form of international tin agreement, general trends in world tin production of tin production and consumption in the last 50 years, operation of the London metal market in arriving at the tin price, U. S. controls on consumption of tin and tinplate, and the importance of the world canning industry.

Advanced Mechanics of Materials, second edition, by Seely Smith. *John Wiley & Sons, Inc.* $8.50, 680 pp., 1952.—The first edition of this book appeared 20 years ago. The current edition takes into account the many advances made and the better preparation on the part of students and engineers for handling of knowledge in this field. The book consists of six parts: Preliminary considerations, special topics on the strength and stiffness of members subjected to static loads, localized stresses—stress concentration, energy methods, influence of small inelastic strains on the load carrying capacity of members, and introduction to instability—buckling loads.

FBI Register of British Manufacturers 1952-53, 25th edition. *Federation of British Industries.* About $6.00, 922 pp., 1952.—The 1951 FBI Register comprises seven sections: Introduction; products section; classification of member firms; advertisements section; addresses section; trade associations; brand and trade names, and trade marks.

ORDER YOUR BOOKS THROUGH AIME—Address Irene K. Sharp, Book Department. Ten per cent discount given whenever possible. Order Government publications direct from the agency concerned.

Textbook of Engineering Materials, by Melvin Nord. *John Wiley & Sons, Inc.* $6.50, 518 pp., 1952.—The book is designed to introduce the engineering point of view early in the student's career. Engineering materials are presented in a fundamental, coherent manner, rather than in an unrelated series. The subject is treated generally in the first half of the book. Fundamental concepts in physics, chemistry, geology and other subjects are introduced at an elementary level. Specific engineering materials are treated in light of the generalizations made, in the second half of the book. Basic concepts are applied to problems of occurrence, production, properties, and use of various materials.

Quantitative Analysis, by Harvey Diehl and G. Frederick Smith. *John Wiley & Sons, Inc.* $5.00, 539 pp., 1952.—The book assumes that the reader has a prior knowledge of qualitative analysis. Theory begins with the solubility principle and calculation begins at a level requiring only a general understanding of chemistry. The book treats several new procedures including the versene method of determining the hardness of water. The work contains more than 450 problems which demonstrate the various types of calculations in stoichiometry and equilibrium problems, illustrate methods not otherwise covered in the text, or show how to apply chemical knowledge to devising methods for the quantitative analysis of various products and mixtures.

Engineering Consultants Directory. *American Institute of Consulting Engineers, Inc.* $2.50, 106 pp., 1952.—This is a compilation of consulting engineers, their fields of specialization, the kind of professional work they are prepared to undertake, and pertinent data regarding each individual and his associates. The information should prove of practical value to those who seek advice and assistance from engineering specialists. A directory of firms is also included, giving information as to the type of engineering work they are equipped to undertake. Wide distribution is planned for the book.

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Industrial Notes

• The new stainless steel walled building atop a hill overlooking the primary steel operations of Allegheny Ludlum Steel Corp., at Brackenridge, Pa., has been dedicated. It is the first unit of what ultimately will be a center containing the company's research facilities. The first unit has been named after Vere B. Browne, retired vice-president and technical director of the company. Research work in the first new buildings will be devoted to corrosion; X-ray; stainless; metallographic; welding and magnetic material sections; high temperature alloys; tool and die steels; and carbides and titaniums. At present the firm has three other research units, at Dunkirk and Watervliet, N. Y., and Ferndale, Mich.

• Defense Production Administration announced that Olin Industries, Inc., have been given the go-ahead to construct a $170 million aluminum plant. The company is expected to produce 110,000 tons of primary aluminum in a year. If the goal is reached, it will represent more than half of the 200,000 tons of added capacity declared necessary for national defense.

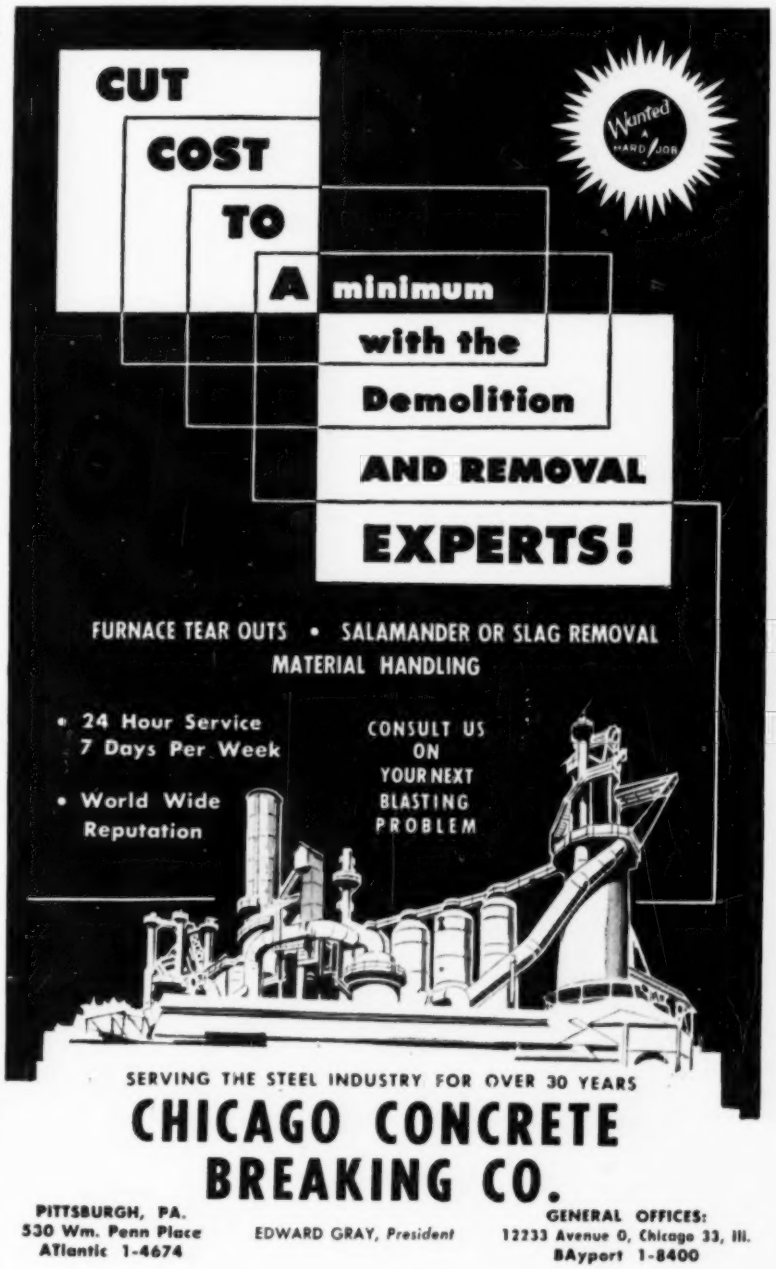
• Aluminum Co. of America's Rockdale, Texas, works, the nation's newest aluminum smelting plant, is in production. R. T. Whitzel, manager of Alcoa's smelting div., and J. D. Harper, Rockdale works' manager, poured the initial aluminum from the first operating pot line. When completed, the installation will include a smelting plant with four pot lines; a plant manufacturing carbon electrodes; a power plant; and a facility for mining lignite.

• The $38 million plant of Armco Steel Corp., is expected to go into production at Middletown, Ohio early in February 1953. The plant is built on a 300-acre tract across from the firm's East works. It will have one of the most modern blast furnaces in the country; a battery of 76 coke ovens and a byproducts plant. A powerhouse and other auxiliary facilities are under construction. The new furnace will supply 1500 tons of pig iron per day when in operation.

• Alloy production was started at Vanadium Corp. of America's $2.5 million plant near Cambridge, Ohio. The installation includes three low frequency induction furnaces formerly used at the company's leased Chester, Pa., plant, since returned to the U. S. Government. Activities at the Ohio plant are currently confined to the manufacture of aluminum alloys. Ferrovanadium, grainal alloys, low carbon ferrotitanium and other alloys are scheduled for production when the plant is completed. A pilot plant for experimental work in aluminum and alloy metal production is scheduled for construction.

• A new organization, Mathematical Computing Service, devoted exclusively to applied mathematics, has been formed. It will render service to industries and universities desiring engineering calculations, charts, and nomographs of a high degree of complexity and the treatment of relating mathematical problems in metallurgical engineering.

• Alumina Ore Co. is planning an addition to its new alumina works at Bauxite, Ark., at a cost of more than $5 million. The new unit would be for manufacture of finished chemical products from alumina processed from Arkansas bauxite.



New $82,000 annealing furnace at Anaconda Wire & Cable Co.'s Anderson mill can heat 6250 lb of copper per hr to 900°F. It is gas fired and radiant tube heated. Copper is loaded on bulk-head type trays and fed into annealer by pusher control.

STEEL men will have many memories of 1952. Some memories, such as the steel strike, will be bitter, but others will stand as a symbol of the might of democracy and its economic system. Perhaps one of the most impressive events of the year took place at U. S. Steel's Homestead works in Pittsburgh, when the firm's billionth ton of steel was poured. No one can hope to measure the benefits that have come from this billion tons of steel. It has contributed to the happiness of many more Americans than can be counted. It has gone into refrigerators, automobiles, bridges, buildings, and countless other things we take for granted. And it has gone into the things that have made the nation safe, the tanks, guns, and ships which have been used to defend those truths we feel to be *self evident*.

Slightly more than 50 years were required to pour a billion tons, but Clifford F. Hood, new president of U. S. Steel, says that the next big development will be speeding the time required to convert iron and other raw materials into finished steel. At the rate U. S. Steel is currently operating, it could pour its second billion tons in 30 years. Today, this one company has roughly the same capacity as all of the Soviet Union.

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REPORTS on free world tungsten steadily become more encouraging, as new discoveries are made and already operating mines expand production. Not so long ago the Chinese Communist Fourth Army marched into Kiangsi and Kwangtung Provinces, cutting off one third of world tungsten ore production. Before the Communist victory, tungsten ore sold at about $2000 per ton of 60 pct concentrate, compared with $6500 today. Exploration and development have been given a tremendous shot in the arm by the price. Free world tungsten production rose from 15,050 to 25,618 tons, in 1951. During the same period production in Communist controlled countries reportedly rose from 15,100 to 24,508 tons. Figures are for tons of concentrates, each containing 1049 lb of tungsten trioxide. U. S. output during the same period rose from 4403 tons to 5693 tons. Thus far, U. S. production has indicated that it will rise again in 1952. For the first half of the year, output reached 3,397,000 lb, compared with 5,869,000 lb for all of 1951.

South Korea has offered indications of growing importance in the world tungsten picture. Reserves have been reported at 2.28 million tons, with an average metal content of 1.7 pct. All South Korean tungsten concentrate is sent to the U. S., under an agreement between the two countries. Canada, never before significant in the tungsten scene, may prove to be another important factor in overcoming the handicap imposed by Communist China. Western Tungsten Copper Mines, Ltd., has announced that it plans to expand milling capacity at Red Nose mine on Rocher de Boule, near Hazelton, B. C., from 150 tons per day to 500 tons. Reserves of 500,000 tons have been disclosed with additional diamond drilling under way.

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STEEL industry leaders have recommended a three part schedule to bring to an end government allotments of steel to civilian users by March 31. The plan is based on the surmise that there will be 31 pct more steel this year than in 1952. NPA received the plan without comment. It has been reported that the government agency feels that decontrol should be a somewhat slower process. A schedule submitted by NPA representatives, however, is quite similar to the one offered by industry.

First step in the schedule proposed by industry is immediate open ending of the Controlled Materials Plan for steel. It would permit steel mills to sell products to buyers who have no priorities, on condition that the mills have room for additional orders at the conclusion of product "lead times" (period before the first day of the month in which delivery is requested).

Since mills are still working on backlogs resulting from the steel strike, it is unlikely that the word immediate is used in its strictest sense. The initial phase might not begin before the first of the year.

The second step in the plan would revoke all outstanding non-military authority for steel under the Controlled Materials Plan. For the beginning of the second quarter, April 1, CMP allotments would be made only for military and atomic energy requirements.

NPA would go on with its directives during this period, telling mills what kind of products to make and the amount of steel to be set aside for military needs. NPA would continue to control warehouse distribution and inventories in the hands of consumers.

All CMP allotments for steel would end around July 1, along with inventory and warehouse control, in the third step of the plan. Remaining would be directives and set asides for military and atomic output.

While not in complete agreement with the plan presented by industry, definite signs of near-accord could be discerned on the part of Government men at a meeting held recently in Washington. Difference between NPA plan and its industry counterpart seems to be merely in timing. NPA would prefer to maintain some kind of allotment system until July 1, or about three months longer. No immediate decison can be expected. All that can be expected now is that serious consideration will be given to the moves required to get Government out of steel.

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AN MIT economist has provided Americans with myth destroying information which should prove effective against the *gentlemen* of the extreme left. For a number of years they have charged that a small number of companies in each industry has been grabbing more and more of the industry's business. M. A. Adelman has shown that the concentration of business in the hands of big corporations has not shown any sign of growth during the last 50 years. Quite the contrary, there has been a slight decline in this concentration, according to Adelman. There are more small companies per thousand population than ever before.

Bearing this out is the fact that International Harvester is now doing 41 pct of the possible market against a former 85 pct; American Can Co. has dropped from 90 pct to 50 pct; American Smelting is down to 31 pct compared with 90 pct; and lastly, U. S. Steel which has plummeted from 65 pct to slightly more than 30 pct of the steel market. Declines of the same proportions have hit big companies in other industries. Price cuts on Detroit's 1953 automobiles indicate that even among the biggest competition is still one of the most important household gods. Big business is still slugging it out for the attention of the American consumer. It's a battle which can only have one result—benefit to the man who buys.

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SAND casting of a magnesium alloy wing section by Aluminum Co. of America for Northrup Aircraft, Inc., created more than passing interest at the 8th annual meeting of the Magnesium Assn., Nov. 10 to 11, 1952, in New York City. The casting was made as part of a research project for the U. S. Air Force. The project was a segment of a long term program for casting and forging large aircraft components.

The wing section, 16 ft long, 3½ ft at its widest point, and with a minimum section thickness of ¼ in., was cast in two sections, using alloy AZ92-T6. Five hundred lb of magnesium was poured for the 200 lb wing. Alcoa's W. D. Stewart gave a limited account of the project, pointing out that security restrictions prevented him from giving a detailed description of casting practices.

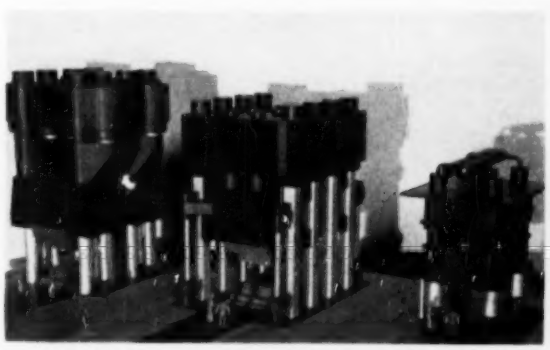
Another interesting matter broached to those attending the session on magnesium castings was the opinion of R. H. Osborne, of R. H. Osborne Mfg. Co., that thin wall magnesium castings offer many advantages over fabricated units, especially for complicated shapes. Cast parts, according to Osborne, can be made more accurately and are more consistent in shape. Pattern tooling for castings is much cheaper than initial tooling costs for blanking dies, forming dies, assembly fixtures, etc. Parts presently sand cast from magnesium are lip intake ducts, pilot window sliding frames, escape hatch frames, air scoops, guided missiles, and airborne radar equipment. Wall thicknesses of 1/16 to 1/8 in. are not uncommon.

A claim that the shell molding process can be used successfully as a supplement to existing magnesium casting practices, was advanced by A. Marotta, of Utica Radiator Corp. He saw advantages to the foundry and customer, although a cost comparison with conventional sand practice has not been made as yet. Shell moldings for magnesium castings were said to offer these advantages:

- Closer dimensional tolerances
- Extremely smooth finish
- Less risers, chills, and gates necessary, reducing metal loss
- Scrap loss reduced because shell molds are easy to handle and do not break or crumble easily
- Increased production per man-hour
- Skilled labor not required
- Molds can be stored indefinitely

Disadvantages to be considered are difficulty in shaking out or removing the shell mold from a magnesium casting, and disagreeable odor after pouring.

More than 100,000 tons of magnesium are being produced annually, with consumption at about 48,000 tons per year—12,000 tons used commercially, and 36,000 going to defense or rated projects. Stockpile requirements take the difference between production and consumption. Production is currently at about half of the World War II peak. Consumption is approximately one third that of the same period (1943 to 1944).



U. S. Air Force officials and Aluminum Co. of America have signed a contract which will result in two of the world's largest forging presses going into construction at Alcoa's Cleveland works. The 50,000-ton press and the 35,000-ton models on the left are contrasted with a 15,000-ton press now in operation at the Cleveland works. The giants are expected to be in operation by 1954.

Heroult

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New York 18, N. Y.

Drift of Things

IKE'S amiable expression and apparent sincerity impressed AIME members as did the theme of his talk. Cautioning against the gradually increasing power of the federal government, he said: . . . "freedom as we know it is in no greater danger from without than it is from our own lack of understanding of this creeping paralysis from within." Next February, it will be three years since he uttered these words at Annual Meeting welcoming ceremonies, but the theme was oft repeated in his winning campaign. This brush with the exalted, brief though it was, makes Washington look friendlier to many AIME members than it has for years.

In addition, and probably more important to AIME, is appointment of George M. Humphrey, member since '22, as Secretary of the Treasury. Born and raised in Michigan, Mr. Humphrey went on to Ann Arbor to study law. Three years after joining the M. A. Hanna Co., and when only thirty, he became a partner. Today he is chairman of the board but the record of his other accomplishments such as the organization of National Steel Corp. in 1929 and Pittsburgh Consolidation Coal Co. in 1945 indicates that he has blended law books with balance statements well. Younger blood remembers his name most in connection with the Labrador-Quebec iron ore deposits. Mr. Humphrey helped form the Iron Ore Co. of Canada, and became its first president. Labrador-Quebec is taken quite for granted today as one of the promising new sources of ore, however, it did not get that way without a great deal of selling. Mr. Humphrey and others had to have the courage to back up a difficult, but good, engineering bet.

FROM the business man's viewpoint, it was reported at the annual meeting of NAM, the election resulted in a new and hopeful opportunity to show what business really looks like when not villanized by hostile government. And the President-elect has given business a square shake in his selection of advisers. Now that the public is getting another chance to see what free enterprise can do, business must put its best foot forward.

An honorable end of the Korean war is naturally the most-to-be-hoped-for achievement of the administration. We concur, but there are some things which we hope will be given high priority on the new administration's list of things to be done. Business has long claimed that redistribution of the tax load can be done in a way to encourage business growth and the presumption is that more business means more sources of tax revenue. Secretary Snyder was always willing to listen to such proposals but usually dampened enthusiasm for studies of tax reform when he said in effect: any tax reforms will be considered so long as they produce the same revenue. Mr. Humphrey will have a tough time with this one particularly since a large percentage of the short term debt will become due in the next four years and defense expenditures will be difficult to cut.

Then there is the State Dept. Conversation of mining men overheard in a Lima, Peru, hotel lobby led us to believe that the prestige of the U. S. in the Western Hemisphere has dwindled to a very low ebb indeed. Apparently a mine manager representing a U. S. firm in Bolivia had been incarcerated on trumped-up political charges by the local government. The man happened to be a British subject and his government had him released in short order. Concensus among U. S. citizens: that he was fortunate to be British not Yankee (or he would still be in jail). Apparently being a citizen of the U. S. no longer carries the prestige and security abroad which it did. In this era, when the role of the U. S. in world affairs is in the ascendency, it is more important than ever that our citizens abroad be protected.

Investigation of business by government could be a little more responsible. For instance, Mr. Hoover of the FBI conducts his business in a quiet, thorough, and nonpolitical manner. There have been cases where the Government used notoriety and flamboyance in its prosecution of corporations—to make political hay regardless of verdict.

WE had a tough time getting wrappers big enough to carry this three-section mailing of the journals. All AIME members should have received a magazine, the index of the 1952 Journal, and a copy of the *Yearbook and Directory*. Directories were not sent to students or nonmember subscribers. In thumbing through the Yearbook section, we were impressed by the tremendous compilation of historical and current data on AIME which was provided by the Secretary's office. Certainly a great number of people are taking and have taken active parts in the affairs of the Institute.

The listing of members in the Directory section is both alphabetical and geographical. In 1950 an experiment with a geographical-company listing of members was undertaken in the Directory of that year, but the Council of Section Delegates reported it to be confusing and of less value than the straight geographical listing.

A new method of keeping the Directory data has been used. It is called Flexoprint and was developed by Remington Rand. Names, titles, and addresses as they appear in the Directory have been typed by electric typewriter on cards which are inserted in frames. Each frame represents a page. The frames are photographed and printing plates made from the negative. As changes of position or company listing are received at headquarters, they will be immediately typed on cards and inserted in their proper position in the frames. When a new Directory is wanted, it is only necessary to make the printing plates by photo-offset. No special preparation is required or proofreading. The entire process, including amortization of investment for the system, will lower the cost of Directories.

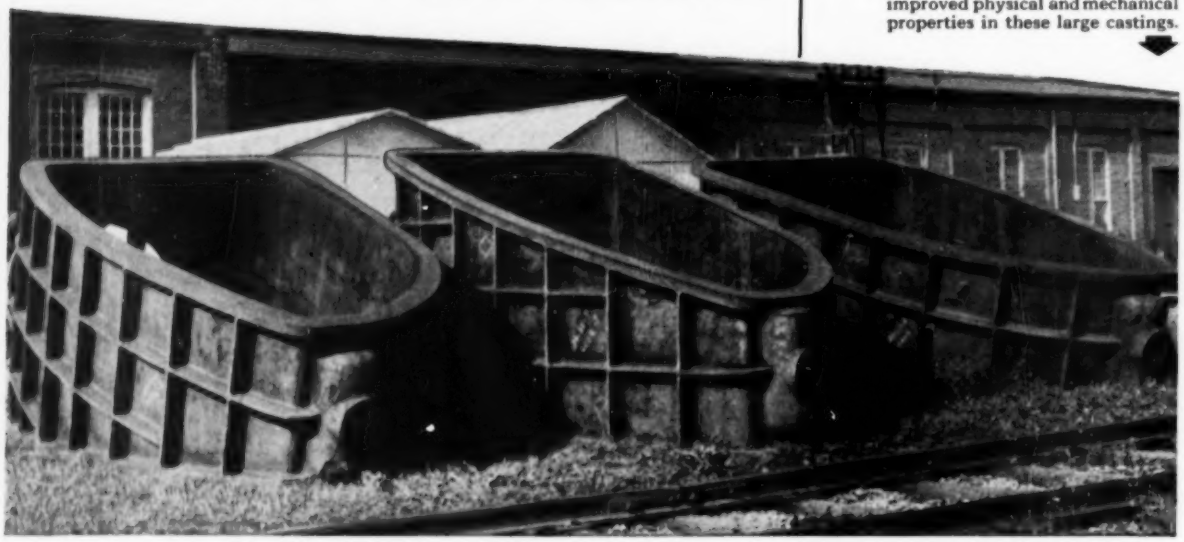
Following the names listed in the geographical section are code letters. Once you become familiar with them (and it takes some doing) they are quite useful. They indicate which of the ten Divisions of AIME a member belongs to. You are entitled to belong to as many as you wish, hence the multiplicity of letters after each name. For the key to the code turn to page 233 of the Directory. The system reminds us of the remark made by a perplexed student of descriptive geometry: "It's not the intrinsic difficulty of the problem which bothers me, it's multiplicity of detail."

FOR about four years we have been carrying on a continuous readership study of the magazine and the editors have long been impressed by the fact that the "Drift of Things" as followed by E. H. Robie has consistently ranked high in readership among the features. Further evidence of the excellence of the product of his portable typewriter is manifested by the thick folder of correspondence in our files marked "Comments on Drift." The growth of the Institute in recent years both in membership and activities has multiplied Mr. Robie's work to the point that he has decided he can no longer continue "Drift." We will try, however, to persuade him to step in as a guest writer at every opportunity.

John V. Beall

NICKEL CAST IRON PROVIDES HIGH STRENGTH AND PRESSURE TIGHTNESS...

Water boxes produced of nickel cast iron by Kutztown Foundry & Machine Corp., for Foster Wheeler Corp., to obtain greatly improved physical and mechanical properties in these large castings.



IN CASTINGS OF LARGE DIMENSIONS

Here are three water boxes, weighing 21,500 pounds each, for a power-plant condenser system designed by Foster Wheeler Corporation, New York City.

Excessive pressures of the service involved call for high strength, as well as high density of grain structure.

Accordingly, these water boxes, produced by Kutztown Foundry & Machine Corporation, Kutztown, Pa., were cast in 2% nickel cast iron.

Meeting ASTM "Class 50" specification (minimum 50,000 psi tensile strength) and characterized by dense structure with fine dispersion of graphite throughout, this nickel cast iron provides an extraordinary degree of pressure tightness under hydrostatic pressures.

The matrix of nickel alloyed iron closely resembles the pearlitic matrix found in high-carbon steels. In contrast, the matrix of ordinary plain iron resembles that found in low-carbon steels.

Throughout industry, nickel cast irons spell economy when you need strength, wear resistance and pressure tightness. Write for our suggestions regarding the best nickel alloyed iron for your specific applications.

At the present time, the bulk of the nickel produced is being diverted to defense. Through application to the appropriate authorities, nickel is obtainable for the production of engineering nickel cast irons for many end uses in defense and defense supporting industries.

The International Nickel Company, Inc.
Dept. 20, 67 Wall Street, New York 5, N. Y.

Please send me booklet entitled, "Guide to the Selection of Engineering Cast Irons."

Name _____ Title _____

Company _____

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THE INTERNATIONAL NICKEL COMPANY, INC. 67 WALL STREET NEW YORK 5, N.Y.

Interchange of ideas and practices in the use of rare earth as additions to steel remains limited. Considerable work must be done before an evaluation of the effect and economics of rare earths can be reached. Major steel companies have said little, while small firms seem reluctant to assume the expense of pilot plant spade work. However, rare earth consumption continues to rise, and it seems certain that the price will decline.

Rare earths can be used in acid electric furnace practice if precautions are taken against contamination with slag. One foundry uses rare earth additions where specifications call for high impact strengths at extremely low temperatures and cost is not a factor.

U. S. steel manufacturers spent an estimated $320 million in foreign countries last year in payment for iron ore, nickel, palm oil, and other materials, metals and ores. Canada got the lion's share of the foreign spending, with $91 million going for nickel, iron ore, zinc, ferromanganese and other ferroalloy materials.

Intensification of civic atmosphere polution programs has made electric furnace fume control critical. While no one procedure seems to offer a final solution, work progresses along several different approaches. An industry-wide cooperative project has been suggested to correlate developments.

Shell molded parts offer price savings in contrast to sand castings where surface finish and dimensional tolerances obtained result in reduced machining and finishing costs. One foundry has found in using the process that the as-cast shell molded part usually costs 15 to 20 pct more than a sand casting. Pattern cost is high, and hand operated shell molders are used because of cost of automatic machines.

Reynolds Metals Co. has expanded aluminum production capacity at its Longview, Wash. plant 67 pct by "stretching" each pot in the plant's three potlines. Pots were cut, new sections inserted, and then rewelded. They were made 2 ft longer and 2 ft wider, but still fitted original space. A total of 372 pots were enlarged, with $8 million saved in the process.

No universal human relations program can be evolved that will benefit every plant, and no plan can be successful unless promoted by a sincere management, according to Rudy Smith, of CF&I. Each approach must be designed with individual plant peculiarities in mind.

Kaiser Steel Co. expects to complete expansion of major facilities for the $65 million enlargement of its Fontana, Calif., facility. The program calls for construction of the third Pacific Coast blast furnace, 90 new byproduct coke ovens, a ninth open hearth furnace and major additions to rolling mills.

Two new alloys were announced by Aluminum Co. of America. Designated XA54S and XC56S, their main advantage is that for certain construction use, sections can be half the thickness of other aluminum alloys, yet have the same strength. The alloys are also said to be highly corrosion resistant.

Advertisement

ELECTROMET Data Sheet

A Digest of the Production, Properties, and Uses of Steels and Other Metals

Published by Electro Metallurgical Company, a Division of Union Carbide and Carbon Corporation, 30 East 42nd Street, New York 17, N. Y. • In Canada: Electro Metallurgical Company of Canada, Limited, Welland, Ontario.

COLUMBIUM and TANTALUM

Strategic Combination for Imparting Strength and Stability to High-Temperature Metals

Gas turbines for jet-aircraft engines, and for other similar engines being developed for marine and railway transportation, have greatly increased the demand for high-temperature metals. The present most satisfactory metals are either iron-base, nickel-base, or cobalt-base. They are used in the form of castings and hot-worked products, such as forgings, bars, and sheets.

Many of these special metals contain columbium because of its beneficial effect on high-temperature strength in both cast and wrought products. Investigations have shown that columbium is one of the key alloys for imparting high-temperature strength and stability in metals suitable for operating temperatures up to 1500 deg. F. and above.

Need for New Alloy

Originally, a ferrocolumbium alloy containing approximately 55 per cent columbium and 5 per cent tantalum was employed in the production of many of these high-temperature metals. However, with the increased use of high-temperature metals and columbium-bearing 18-8 stainless steels, the demand for columbium exceeded its availability.

ELECTROMET's Research Laboratories began investigations to ascertain whether an alloy containing more tantalum and less columbium would be equally satisfactory for producing the high-temperature metals. Columbium and tantalum alone, as well as combination alloys of columbium plus tantalum, were tested.

Results of Tests

The alloy N-155 was selected for tests. It is an iron-base alloy with the following approximate analysis:

Chromium	20 per cent
Nickel	20 per cent
Cobalt	20 per cent
Tungsten	3 per cent
Molybdenum	2 per cent
Columbium	1 per cent
Nitrogen	0.15 per cent
Carbon	max. 0.35 per cent

The data in the table below describe the mechanical properties, at room temperature, of this low-carbon N-155 alloy modified with columbium and tantalum alone, and with combinations of columbium plus tantalum. The metal modified with columbium has good strength and high ductility at room temperature. These same good properties are obtained when tantalum, or tantalum plus columbium, is substituted for the columbium.

Stress-to-rupture tests were also conducted on these same modified low-carbon N-155 alloys at 1350 and 1500 deg. F. The data show (see table) that when the columbium is replaced with a mixture of columbium plus tantalum, the strength of the metal remains substantially unaffected at 1350 and 1500 deg. F. Also, when all of the columbium is replaced with tantalum, the strength of the metal at 1500 deg. F. is equivalent to that obtained with columbium. Hence, from the standpoint of high temperature strength, columbium and tantalum can be used interchangeably, or in combination.

Help to Industry

ELECTROMET has developed an alloy containing approximately 20 per cent tantalum and 40 per cent columbium for use in high-temperature metals and stainless steels. It is known as ELECTROMET ferrotantalum-columbium.

Industrial experience with this comparatively new alloy has confirmed the favorable results of the experimental work. The alloy has already aided considerably in augmenting the supply of columbium alloys, since it is just as effective as ELECTROMET ferrocolumbium, with 50 to 60 per cent columbium, for giving strength at high temperatures.

Furthermore, the new alloy is now preferred because of its greater availability. It should be added to a thoroughly deoxidized metal bath to obtain the best results. A recovery of about 90 per cent for the columbium and 80 per cent for the tantalum may be expected.

For further information regarding alloys for high-temperature metals, write to the nearest ELECTROMET office: in Birmingham, Chicago, Cleveland, Detroit, Houston, Los Angeles, New York, Pittsburgh, or San Francisco. In Canada: Welland, Ontario.

Properties of Modified N-155 Alloys Compared*

Typical Analysis, %**	With Columbium	With Columbium and Tantalum		With Tantalum
Columbium	1.13	0.58	0.49	....
Tantalum	0.08	0.64	0.53	1.47
Carbon	0.12	0.13	0.13	0.12
Nitrogen	0.13	0.14	0.14	0.14
At Room Temperature				
Tensile Strength, psi	119,000	123,200	117,500	122,100
Yield Strength, psi	56,700	59,500	52,000	59,100
Elongation in 2 in., %	52	47	57	54
Reduction of Area, %	69	65	69	56
At 1350 deg. F.				
Stress to Cause Rupture, psi				
In 100 hr.	31,000	34,000	33,500	....
In 1000 hr.	23,000	25,000	23,000	....
At 1500 deg. F.				
Stress to Cause Rupture, psi				
In 100 hr.	20,000	20,000	20,500	20,000
In 1000 hr.	15,000	14,000	15,500	15,000

* Tests made on standard samples from one-inch round bars, water-quenched from 2250 deg. Fahrenheit.
** Composition of the base alloy is given in the text.

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Chuquicamata

A Story of Chilean Copper

LONG before Columbus discovered America the original inhabitants toiled in the copper workings of the Andean Cordillera. Their best diggings appear to have been at Chuquicamata, site of the huge present-day open pit owned by the Chile Exploration Co., subsidiary of Anaconda Copper Mining Co. Located in Antofagasta Province of northern Chile, Chuquicamata is in the Atacama desert, high on the western slope of the Andes at 9500-ft elevation.

In the Chuquicamata area the Atacama desert merges into the Cordillera. The climate is extremely arid and precipitation, either in the form of rain or snow, is very infrequent. The temperature varies from 50° to 85°F during the day, and drops to 20° to 40°F at night.

The Antofagasta and Bolivia Railroad, which connects the Chilean Port of Antofagasta with La Paz, Bolivia, passes within approximately 6 miles of the plant. A branch from the main line to Chuquicamata serves the property for outgoing copper and incoming freight. The distance to Antofagasta by rail is 163 miles.

Previous to the Inca conquest, this region was inhabited by the Chuco Indians, a small tribe, descendants of the Aymaras and Quechuas.

The Incas, whose capital was located in Cuzco, Peru, gradually extended their empire to the south, reaching at one time to what is now central Chile. It is believed that they built crude primitive furnaces on the banks of the Rio Salado where on a small scale they smelted copper ore from Chuquicamata. At the time of the subsequent Spanish conquest, legend has it that Diego de Almagro, on his return to Cuzco in 1536 after his invasion of Chile, obtained from these furnaces metallic copper for horseshoes.

During the period from 1560 to 1879 the high grade veins of the Chuquicamata deposit were worked superficially by the Spaniards and Bolivians. The treaty signed after the war of 1879 recognized the sovereignty of Chile over this territory and Chile, realizing the importance of the district, started to develop it in an orderly manner, although only high grade veins were worked.

New Process Is Key

Reports on the deposit were brought to the attention of Albert C. Burrage, a Boston lawyer and financier, in 1910. Chuquicamata was described as a huge deposit of ore that was not amenable to the usual treatment by concentrating and smelting since much of the copper occurred as oxides. He had been working on a process for the recovery of copper from sulphide ore which involved a sulphating roast, leaching of the roasted product, and precipitation of the copper by means of limestone. Since it appeared that the ore in the Chuquicamata deposit could be beneficiated by his process, he was extremely interested and sent an engineer to Chile to investigate.

At the outset Burrage's representative was able to obtain options on the two principal properties then operating and subsequently options were acquired on numerous other claims in the district. Systematic sampling of all workings was done under his direction. Burrage first recognized the possibility of mining the entire deposit rather than just the high grade areas.

Chilex Is Formed

Since the project was too big for Burrage to finance alone, he made overtures to the Guggenheim interests, who, after a brief period of negotiation, took over Burrage's options toward the end of 1911, and on Jan. 11, 1912 organized the Chile Exploration Co.

Exploration of the deposit by churn drilling was started by Edwin S. Berry and Walter A. Perkins in April 1912. It was soon determined that the extent and depth of the ore were much greater than anticipated.

The progress of the metallurgical engineers under the direction of E. A. Cappelen Smith, consulting metallurgist for M. Guggenheim's Sons, in the development of an economical process for extracting

the copper from the oxide ore, was so successful this firm purchased the property September 1912.

Plans were made to begin large-scale development of the mine and to proceed immediately with the construction of a treatment plant. Actual copper production started in May 1915 and the operations continued under Guggenheim management until February 1923 when the controlling interest was acquired by Anaconda Copper Mining Co.

Not Standing Still

The original plant was designed to treat 10,000 tons of oxide ore daily which would yield approximately 100 million lb of copper annually. Subsequent additions to the plant, the largest of which started in 1925 and was completed in 1927, brought the capacity up to 375 million lb yearly. Additions to the plant as well as improvements in the metallurgical process, together with a higher grade of ore, brought the capacity to 490 million lb per year by 1941. In 1942, at the request of the United States Government, additions to the plant and more equipment for the mine yielded a further production of 50 million lb annually giving a total capacity of 540 million lb when operating 335 days per year. At that time it was realized that a production of this magnitude could not be maintained from oxide ore for any extended length of time, due to the decreasing grade and depletion of the oxide ore reserves. The increase, however, was justified to assist the U. S. in obtaining the copper necessary for its war effort.

By 1948 large tonnages of sulphide ore were being exposed in the pit and were by-passed in the mining operations as far as possible. Continued removal of oxide ore exposed greater tonnages of sulphide ore which up to this time could not be treated for the production of copper as no plant was available for beneficiation of this type ore. To overcome this difficulty and still maintain a production of 540 million lb per year, the board of directors authorized the construction of a plant for the treatment of 30,000 tons of sulphide ore per day. The vast body of sulphide ore underlying the projected depth of the open pit insures a source of copper for many years to come.

Open-Pit Mining Operations

CHUQUICAMATA open-pit mine is capable of producing a total of 105,000 tons daily. When the sulphide plant is operating to capacity the distribution of this quantity will be approximately 30,000 tons sulphide ore, 30,000 tons oxide ore and 45,000 tons of waste. At that time the combined capacity of the oxide and sulphide plants will be about 540 million lb of copper per year.

GENERAL:*

Continuous operation since 1915	
Total tons removed from pit	523,608,000 tons
Oxide ore	362,911,000 tons
Waste material	160,557,000 tons
Total ore remaining in pit	260,000,000 tons
Oxide ore	120,000,000 tons
Sulphide ore (vast sulphide reserves below pit not included)	40,000,000 tons
Total copper produced	10,212,000,000 lb

* Statistics as of Sept. 30, 1952

MINE PRODUCTION:

(when sulphide plant is complete)	
Total tons mined daily	105,000 tons
Sulphide ore	30,000 tons
Oxide ore	30,000 tons
Waste material	45,000 tons

Most rock is moved by rail. Several electric engines have Diesel auxiliaries for operation away from sources of power.

The Story of

As the well known oxide deposits at Chuquicamata were depleted, steps were taken to utilize the vast sulphide ore reserves that began to be uncovered. The result was the construction of a new modern sulphide concentrator and smelter at the site of one of the largest known copper orebodies. This project, of tremendous magnitude, will stimulate the thinking of engineers, designers, and economists for a long time to come.

The story of Chuquicamata presented here is a condensed version of the complete story featured in the December 1952 issue of MINING ENGINEERING. Emphasis has been placed on the developments that it was felt would be of special interest to readers of JOURNAL OF METALS, and provide a general background of the overall picture.

The original articles and cooperation of the officers and operating personnel of Anaconda Copper Mining Co. and Chile Exploration Co. are gratefully acknowledged: D. M. Dunbar, Assistant to Executive Vice-President, Chile Exploration Co., "History of Chuquicamata Copper;" V. D. Perry, Chief Geologist, Anaconda Copper Mining Co., "Geology of the Chuquicamata Orebody;" G. S. Wyman, Mine Superintendent, and L. E. Fish, Assistant Mine Superintendent, Chile Exploration Co., "Open-Pit Mining;" Wilbur Jurden, Chief En-

General view shows new sulphide plant, concentrator in foreground, smelter in distance at left.

Sulphide Ore Plant Design

PRIOR to 1925, the practice in designing copper plants was to locate the various units of the reduction works at the most advantageous points of the existing terrain with little regard for tramming or other auxiliaries, and then connect these various units by the essential trackage, conveyor systems, piping, etc., as the need developed. This occasionally led to undesirable track arrangements, especially when it became necessary to extend various portions of the plant. Conveyor systems also became complicated as well as such items as plumbing and electrical distribution.

These conditions were all taken into consideration in the layout of the Andes Copper Mining Co. plant in 1925. This plant, completed in 1927, had parallel buildings, straight and level trackage, and well designed piping and electrical work. However, the buildings were fitted to the existing terrain, instead of changing the terrain to fit the buildings. At Morenci, in 1939, all desirable features of the Andes plant were incorporated and in addition, the terrain was made to fit the reduction works. The success achieved by design methods used influenced and guided the layout of the Chuquicamata sulphide plant, the largest yet conceived.

At Chuquicamata, the problem was further complicated. The choice location for the smelter was undesirable in that it would allow smelter gases to blow into the open-pit mine where 1550 men are employed. The most difficult problem was to provide for receiving various ores both by rail and conveyor: 1—Sulphide bearing residue from the stockpile from which oxide copper had been leached; 2—sulphide bearing residue direct from the leaching vats; 3—sulphide ore crushed at the existing crushing plants and hauled to the concentrator in cars; 4—sulphide ore from the new crushing plant adjacent to the concentrator; and 5—sulphide ore obtained from future underground mining and crushed in the new crushing plant.

Ample space has been provided to allow for future plant expansion without interference to existing operations.

Initially, all ore will be hauled into the plant by rail. Later, a conveyor system from the crushing plant will be installed permitting ore delivery by conveyor and rail simultaneously. The crushing plant itself will originally receive ore by rail, and later by rail and from underground. All new installations, conveyors, reverberatory furnaces, converters, etc. either in the concentrator, smelter, or power plant, can be made without any interference to operations.

The plant is designed to consist ultimately of 24 grinding units having a nominal capacity of 60,000 tons per day, 8 reverberatory furnaces, and 9 converters. Ten grinding units, 4 reverberatories, and 4 converters are being installed under the present program.

Equipment and supplies presented a major problem as everything had to be transported by rail and water, pass through customs, and be erected in a desert country at high altitude over 5000 miles from the source of manufacture.

Chuquicamata

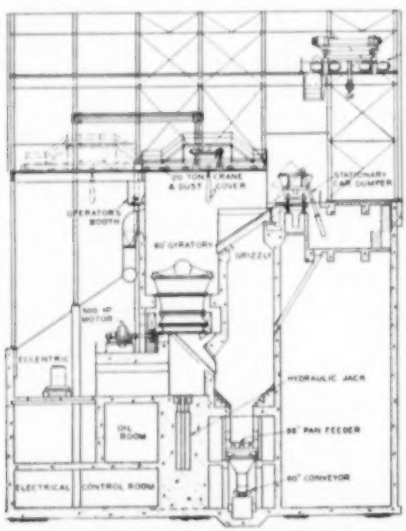
gineer, Anaconda Copper Mining Co., "General Design Sulphide Ore Plant;" A. P. Svenningsen, Chief Design Engineer, Anaconda Copper Mining Co., "Crushing Section;" E. F. Raffo, Design Engineer, Anaconda Copper Mining Co., "Concentrator Design;" D. S. Sanders, Superintendent, and E. W. Witcomb, Assistant Superintendent of Concentrator, Chile Exploration Co., "Concentrator Operation;" R. M. Kuralt, Design Engineer, Anaconda Copper Mining Co., "Tailing Disposal;" H. G. Dwyer, Design Engineer, Anaconda Copper Mining Co., "Smelter;" Clarence W. Dunham, Consulting Structural Engineer, Anaconda Copper Mining Co., "Smelter Stacks;" J. P. Manning, Jr., Design Engineer, Anaconda Copper Mining Co., "Piping;" Stanley F. French, Chief Electrical Engineer, Anaconda Copper Mining Co., "Electrical Distribution;" B. F. Koch, Steam Power Engineer, Anaconda Copper Mining Co., "Waste-Heat Power Plant;" W. E. Rudolph, Chief Engineer, and R. E. Baylor, Assistant Chief Engineer, Chile Exploration Co., "Water Supply;" E. V. Herring, General Superintendent, and J. C. Allen, Assistant General Superintendent Oxide Plant, Chile Exploration Co., "The Oxide Plant;" A. J. Chellew, Port Agent, and Robert Condit, Assistant Port Agent, Chile Exploration Co., "Antofagasta Copper Yards and Shipping."

Editor, JOURNAL OF METALS

Sulphide Plant Crushing Section

IT was initially planned to use existing crushing facilities with a few additions for both oxide and sulphide ore. However, studies and discussions with plant operators made it evident that a complex problem existed. Sulphide, oxide, and mixed ores from the open-pit mines had to be classified and separated, so that each ore was given the proper processing and treatment. Also involved were storage and transportation of the various ores.

When in 1951 the number of grinding units in the concentrator was increased from seven to ten, the smelter increased proportionally, and therefore the overall tonnages of ore to be handled increased. The building of new crushing plants to handle sulphide ore exclusively thus became technically, as well as economically, advantageous. The company was authorized to construct new crushing plants to handle 30,000 tons of ore per day, and capable of reducing the run-of-open-pit ore to the proper size feed for the 10x14-ft rod mills in the concentrator.



Novel feature of the gyratory unit is the combination 20-ton crane and dust cover. The operator, from his station, will be in full control of all units. Heavy concrete protects the floor supporting ore, and seal prevents dust leakage into the room.

The large size of ore delivered from the open pit, sometimes in pieces as large as 6 to 7 ft diam, required the use of a 60 in. gyratory crusher as the primary breaker. This crusher will have a capacity in excess of 30,000 tons per day. It will be a single discharge unit driven by a 500 hp electric motor through a tear coupling and a floating shaft.

The secondary crushing plants will have three 7 ft standard Symons cone crushers and six 7 ft short head Symons crushers. Between the primary and secondary crushing plants a coarse ore bin will be constructed with a nominal draw-off capacity of 30,000 tons of ore. The coarse ore bin is a large pile of ore having a superstructure supporting the conveyors feeding ore to the bin, and a concrete substructure with 12 openings through which the ore is drawn by 48-in. pan feeders onto the conveyors leading to the secondary crushing plants. Each of the three 7 ft Symons cone crushers will be fed by a 48-in. belt conveyor. To insure a constant and continuous rate of feed from the ore bin, variable speed manganese steel pan feeders, four for each crusher, will be employed.

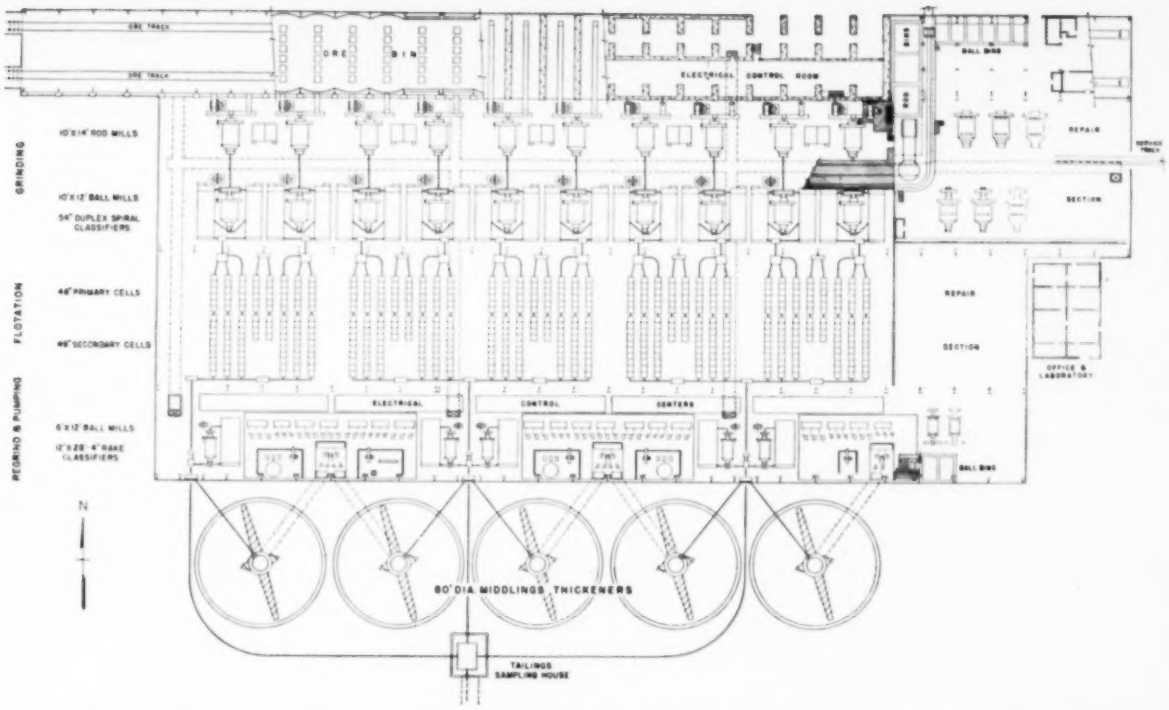
All the crushing plants and the coarse ore bin are interconnected with conveyor belts for transporting ore to the crushers at the desired tonnage. The final product of the new crushing plants is produced by the short head crushers, and will be delivered by conveyor belt to the top of the fine ore bin in the concentrator.

Sulphide ore brought to the primary crushing plant in 40-cu yd (90 ton) capacity side dump railroad cars, will be discharged directly over a grizzly. This is a novel feature in that all crushing plants handling ore as big as the Chuquicamata ore, dump into an ore pocket, where the ore slides onto the grizzly. Since the ore discharge from the cars will be completely under the control of the operator, an ore pocket was not considered necessary. However, the grizzly is of extra heavy design, with a thick manganese steel apron placed over the upper part to protect against impact of large pieces of ore.

A small 20-ton crane is provided for removing or pushing through the large pieces of ore that occasionally lodge in the crusher. In the primary crushing plant this crane has also been transformed into a complete dust cover for the top of the crusher enclosure. From his station, the operator will be in full control of the dumping of the ore cars, the travels of the bridge and trolley of the 20-ton dust cover crane, the lowering and raising of the special hook for breaking jams, the starting and stopping of the 60-in. gyratory, and the starting and stopping of the dust control system.

Automatic bag type dust collectors are being provided at the new primary and two secondary plants. In the primary crushing plant, the dust filters are located inside the building. An exhaust fan with a capacity of 65,000 cfm will pull air through the opening above the grizzly, at a velocity of approximately 100 fpm. Air will also be drawn at the discharge end of the 96-in. pan feeder. Similar arrangements are provided for the secondary crushing plants and at all transfer points from one conveyor to another. Dust control systems for the secondary plants are located outside the building proper.

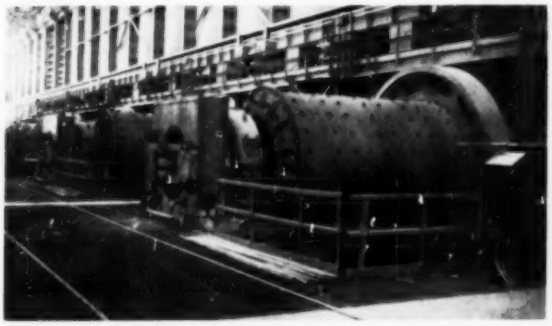
It is expected that the crushing plants, now under construction, will be in operation during the summer of 1953.



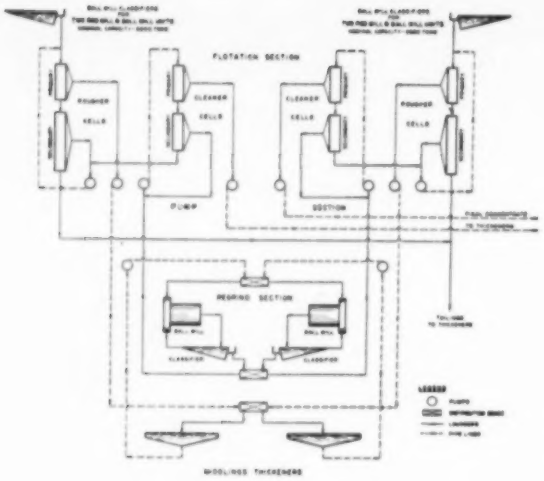
The concentrator was located so it could be supplied efficiently with ore from existing crushing plants and the new crushing plant. Grinding and flotation tests conducted at the Anaconda and Andes concentrators determined the equipment to be used. An arrangement of equipment with adequate auxiliaries, all made up of semi-independent sections, was worked out so original mill capacity could be increased without affecting existing facilities and operation.

Concentrator Design and Operation

NUMEROUS grinding and flotation tests at the Anaconda and Andes concentrators under actual operating conditions helped to establish the major equipment best suited for treating the Chuquicamata ore efficiently, while preliminary design layouts were still being made. While design has been based primarily on the treatment of sulphide ore crushed to about ¾ or 1 in., until the new sulphide crushing plant is completed it will be necessary to handle ore crushed to ⅜ in. in the existing oxide crushing plants, and some mixed ore with oxide copper content removed by leaching. This mixed ore, known



A combination of 10x14-ft rod mills, operating in open circuit, and 10x12-ft overflow-type ball mills in closed circuit with two 54-in. duplex spiral classifiers, provided the most evenly balanced operating arrangement. Above is closeup of the rod mills and the grinding bay is seen at right.



Flowsheet of Regrind Circuit

as leached residue, will contain some sulphuric acid and will require special protective measures.

The principal sulphide copper mineral is chalcocite with varying amounts of covellite, enargite, and chalcopyrite. The oxidized portion is principally antlerite, a green basic copper sulphate. The gangue minerals include quartz, sericite, pyrite, feldspars, and kaolin. Chuquicamata pit ores have proved to be hard grinding. The results of the flotation and metallurgical work prove that good extraction and concentrate grade can be obtained under a rather wide range of conditions.

The leached residue, as well as the sulphide ore crushed at the existing oxide plant, is hauled by rail in 60-ton side dump cars over the fine ore bin. When the new sulphide crushing plant north of the concentrator is completed, ore will also be delivered by conveyors. The ore bin was designed for an approximate drawable storage of a nominal day's supply—about 2500 tons of dry ore per section. A rectangular bottom bin with replaceable vertical steel walls was determined to be most suitable and easiest to protect from acid. Asphalt mastic lining was placed on the bottom and along the sides where not subject to wear by moving ore. As further precautions, the bin bottom was sloped to a series of lead pipe drains, and acid resistant stainless steel used to line feeder openings in the bottom of the bin.

Grinding tests at Anaconda with 9x12 ft rod mills in series with Hardinge ball mills determined that two stage grinding would be used at Chuquicamata. It was decided that a 10x14 ft rod mill operating in open circuit, and a 10x12 ft overflow type ball mill operating in closed circuit with two 54 in. duplex spiral classifiers, would provide the most evenly balanced operating arrangement.

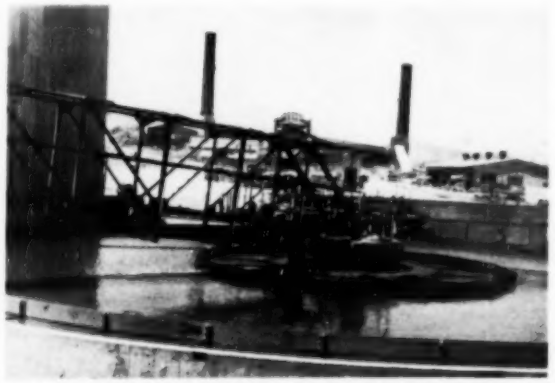
As the ore enters the rod mill through the feed chute, water and lime are introduced through a funnel arrangement that can be observed by the operator. Milk of lime is added to obtain the maximum benefit of mixing with the ore. This protects the equipment from corrosion due to entrained acids and soluble copper salts. Lime consumption is 3 to 5 lb per ton of ore. The rod mill product is laundered directly to the ball mill scoop box where it combines with the classifier sands. The primary promoter reagent, Aerofloat 238, is added to the 10 mesh discharge of the rod mill as it is laundered to the ball mill scoop box. In anticipation that it might prove advantageous in future operation to launder the rod mill product directly to the classifiers, an adequate drop was provided between the rod and ball mills to permit this. Classifiers are arranged with one duplex on each side of a ball mill. Ball mill discharge is divided by a specially designed splitter to assure an equalized load on each classifier. Rod and ball mills are driven by identical 800 hp synchronous motors.

In general the metallurgy of the ore requires a fairly close control of the grind to the primary rougher flotation, and of the regrind of the primary rougher concentrate before cleaning. Regrinding is necessary to reduce the insoluble content of the final concentrate since a large part of the middling combinations are between the copper and silica minerals. The alkalinity of the pulps can vary between wide limits and control of the percent solids gives the desired grind.

The classifier overflow is regulated to produce a grind of about 12 to 15 pct +65 mesh and 60 pct −200 mesh. A secondary promoter Z-4 (aml zanthate) and the pine oil frother are added in the overflow launder where a pH of 10 to 11 is maintained. Comparatively heavy beds of froth on the rougher cells minimize the adjustments of air and pulp levels.

The overflow from the two classifiers in each grinding section is collected in a common junction box and laundered directly to the flotation section, where it is sampled as it enters the rougher flotation distribution box. The rougher flotation section for each grinding unit is made up of three machines of 3 two-cell primary roughers and 5 two-cell secondary roughers. Only one cleaner machine, consisting of 3 two-cell primaries and 2 two-cell secondaries, is provided for each of the grinding sections. The flotation cells are all Agitair machines using mechanical and air agitation. Space has been provided for an additional rougher machine and an increase in cleaner capacity if needed. The froth from the primary rougher cells of two sections is combined and thickened for feed to the regrind circuit. New water, and milk of lime for required alkalinity, are added ahead of the ball mill. The classifier overflow at an approximate 200 mesh grind and 9.0 to 9.5 pH is pumped to cleaner flotation, and is split equally to two cleaner sections.

All flotation products except tailings are laundered directly to pump sumps located below the



One 80-ft middling thickener is provided for two mill sections; flexibility is achieved as any of four adjacent mill sections can operate with either tank serving that group.

Partial view of Chile Exploration Company's open-pit copper mine at Chuquicamata. Over 500 million tons of oxide ore and waste have been removed since 1915. The new sulphide plant is now beginning to process the vast reserves of sulphide ore, and, when operating fully, will treat 30,000 tons of ore per day.

Chuquicamata

Open-pit mine

Sulphide Plant

BEFORE the new sulphide plant went into operation late this year, only the oxide copper ore from the Chuquicamata orebody was processed into copper. This has been mined since 1915, but as mining progressed in depth, mixed oxide-sulphide ore was disclosed as well as the mother lode—a vast sulphide copper orebody of undetermined depth. Sulphide ore was by-passed or stockpiled for the day when a treatment plant would be built. The mixed ores were leached of oxide copper and the residue, containing sulphide minerals was stockpiled.

Today, the giant new sulphide plant is in operation and is expected to treat up to 30,000 tons per day of sulphide ore. The oxide plant will continue to treat oxide ores for many years to come but at reduced rate.

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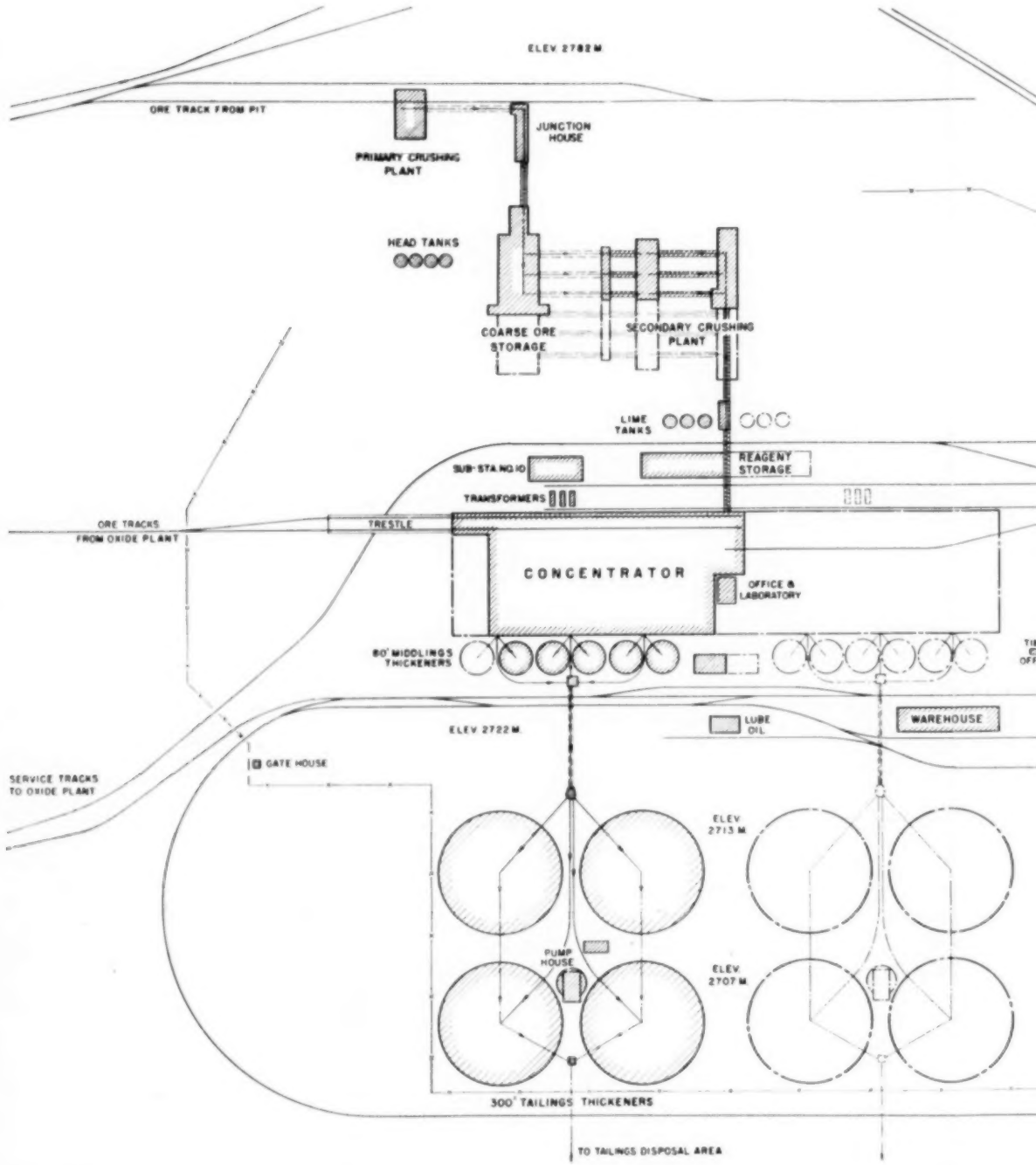
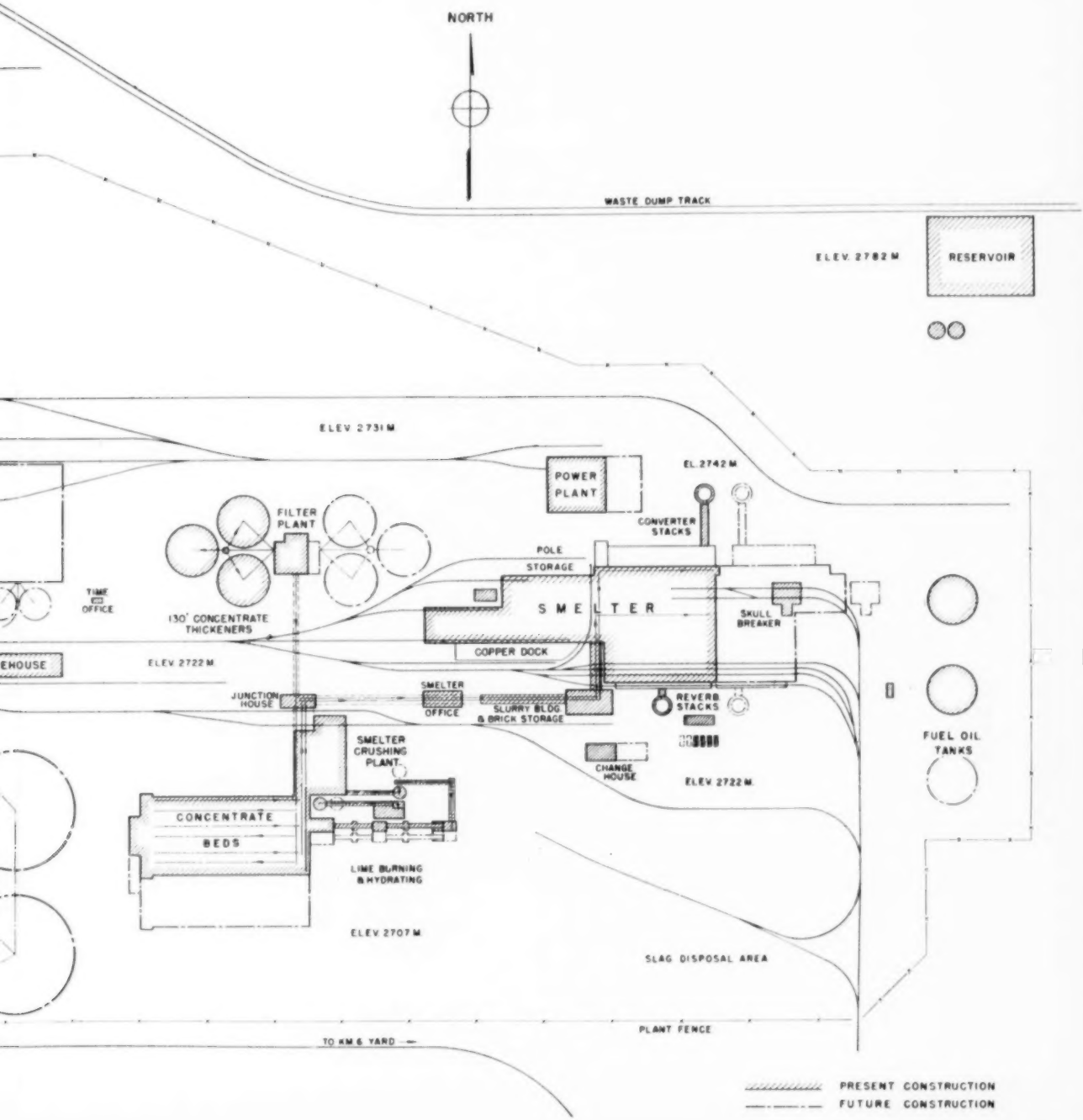


Sulphide Plant Flowsheet

Sulphide ore from the open-pit mine averaging 1.75 pct Cu arrives by rail at the crushing plant where it is reduced to $^3/_4$-in. size and transported to concentrator storage bins. The concentrator consists of 10 sections. In each section ore is reduced to 10 mesh in one 10x14-ft rod mill. The product goes to a 10x12-ft ball mill in closed circuit with two 54-in. spiral classifiers between which the ball mill product is split. Classifier overflow, 14 pct on 65 mesh goes to flotation. For regrinding, the rougher concentrate is thickened and pumped to a 6x12-ft ball mill in closed circuit with a rake classifier. Flotation tailing is thickened to 45 pct solids and flows by gravity to tailing pond. Overflow of the regrind-circuit classifier, 90 pct −200 mesh, goes to flotation. Concentrate is thickened and filtered. Cleaner flotation underflow returns to the rougher circuit. Filtered concentrate, averaging 40 pct Cu, and representing a 91 pct recovery of the sulphide copper is conveyed to the bedding plant. The concentration ratio is approximately 20 to 1.

Silica, lime rock, dust, and reverts are mixed at the bedding plant with concentrate. This mixture, averaging 37 pct Cu is charged to a 30x125-ft reverberatory furnace at the rate of 600 tons per day. Matte averages 45 pct Cu, 25 pct Fe, and 25 pct S and is tapped into 325-cu ft (50 tons matte) ladles from which it is poured into the 13x30-ft converters. Blister copper, 99.5 pct Cu, is transported in same ladles to holding furnaces for casting. The molten converter slag is poured into the reverberatories. Net result: 450 tons copper per day from sulphide ore.



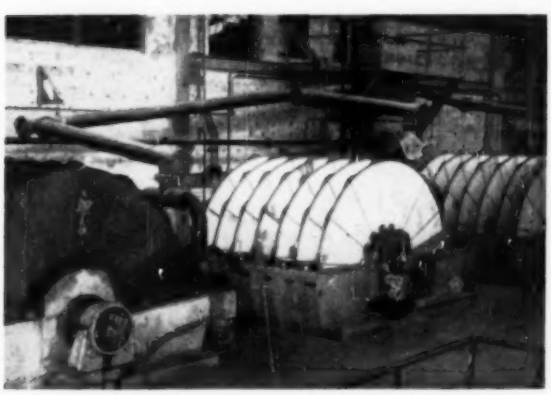
Comparatively heavy beds of froth on the rougher cells minimize the adjustments of air and pulp level.

flotation section. Middling is sampled at the pump sump and then pumped to the middling thickener distribution box. Middling tanks are 80-ft diam torque type thickeners, with one provided for every two mill sections. The distribution box permits any four adjacent mill sections to operate with either of the two tanks serving the group.

The thickened middling is piped in tunnels from directly under the center of the tank to the pump pit for the section it serves, and pumped to a junction box at the regrind section where the pulp flows to the regrind distribution box where it is sampled. Each regrind unit consists of a 6x12-ft ball mill in closed circuit with a 12x28-ft type QSF Dorr classifier. Scott sandwheels, 6 ft diam x 14 in. wide, are attached to the discharge flange of the mills to aid in closing the circuit.

The regrind classifier overflow is pumped to a distribution box in the flotation section ahead of the two cleaner machines included in the grouping of the two sections served by the single regrind circuit. The final concentrate is collected from the primary cleaners and pumped to the concentrator thickener distribution box.

Since about 95 pct of original feed is rejected as tailing, it is important in mill design that the final tailing be collected and laundered to the thickener tanks by gravity. The final tailing from the secondary rougher flotation discharge boxes is sampled and combined with tailings of two adjoining sections. Final samples of tailing are taken at the sampling house before the combined tailing is carried in a single concrete launder directly to the tailing distribution box.



Four 12 ft 6 in. diam, 7-leaf disk filters are housed in the filter plant; only three machines are needed for normal operation, the fourth being for stand-by.

The tailing dewatering system consists of four traction type 300 ft diam thickeners that ultimately will handle tailing from 12 grinding units.

From the concentrator, the concentrate is piped to thickener tanks. These are 130-ft diam torque type thickeners having central and peripheral froth baffles to prevent froth interference at the overflow. The thickened concentrates are then piped to the filter plant. Disk type filters are used in the filter plant. Three of these 12 ft 6 in. diam machines having seven leaves each are required for operation of the initial plant. To maintain production during repair periods, an additional machine was installed.

The final concentrate, after thickening and filtering, is ordinarily bedded for furnace charge, but it can be conveyed direct to the smelter with flux added on top of the concentrate as it passes through the junction house.

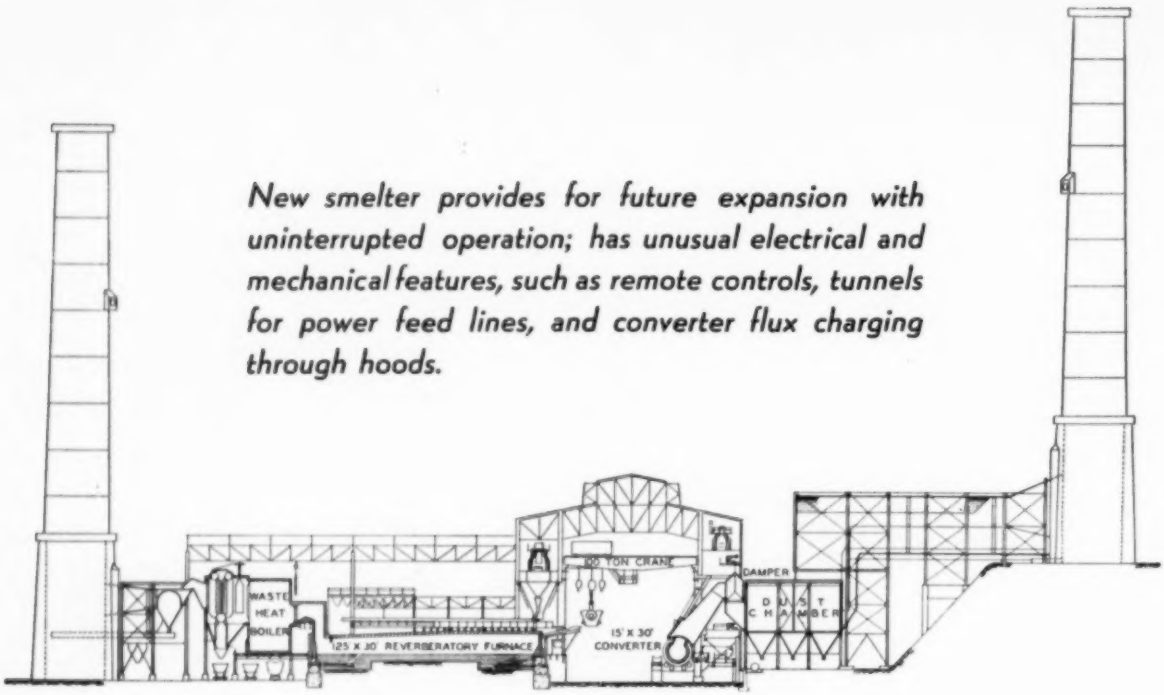
Metallurgy is comparatively simple. Concentrate grade is easily regulated by control of the iron floated. A concentrate of 40 pct is now produced which in turn makes a reverberatory matte containing 44 to 46 pct copper. The concentrate is practically self-fluxing. Lime discard or undersize from the lime burning plant will be used as a basic flux.

The flowsheet provides for the regrinding of the primary rougher concentrate before cleaning, so that the copper minerals therein are liberated from the gangue to make a final copper concentrate of the proper grade and with the least amount of insolubles.

Since the main copper mineral is chalcocite, the problem consists of recovering sufficient pyrite in the final concentrate to provide the smelter with the desired iron and sulphur. Fortunately there is sufficient recoverable pyrite in the ore to permit close control of the pyrite content of the final concentrate.

By the use of small amounts of Z-4 in the rougher and cleaner circuits, a close control of pyrite has been found possible in the final concentrate. Aerofloat 238 has proved to be a good promoter for chalcocite but not for pyrite. The flotation pulp is alkaline and the presence of lime inhibits the flotability of pyrite so that some actual pyrite promoter was found necessary to float more pyrite in the concentrate. By simply replacing a part of the 238 with Z-4, until the desired pyrite content of the concentrate is reached, it has been possible to obtain concentrate of suitable composition.

New smelter provides for future expansion with uninterrupted operation; has unusual electrical and mechanical features, such as remote controls, tunnels for power feed lines, and converter flux charging through hoods.



Smelter

CONSIDERATION for future expansion influenced the design of the new smelter at Chuquicamata. The section of the smelter now going into operation, while large, represents only little more than half of the ultimate plant. Equipment for the direct smelting of raw concentrates has been arranged in the conventional manner although needless details have been eliminated wherever possible while mechanical and electrical features not usually found in copper smelters have been incorporated.

The ultimate smelter will house 7 reverberatory furnaces, 14 waste-heat boilers, 9 Peirce Smith converters, 4 casting furnaces serving 2 casting wheels, and one holding furnace serving a straightline casting machine. For the collection of gases, 2 dust chambers, each with separate stacks, will collect converter gases and 2 balloon flues with separate stacks will collect reverberatory furnace gases. Extensions necessary to complete the ultimate plant will be possible with little interruption to normal operations since the future converters and reverberatory furnaces will be added at the east end while additional casting facilities will be added at the west end of the present plant.

Smelter Crushing Plant

Limerock, converter flux, smelter reverts, and small amounts of other smelter materials are crushed in the smelter crushing plant.

The materials are delivered to the crushing plant in railroad cars and received in a 300-ton track bin. From this bin a 48-in. pan feeder discharges to a 42x40-in. jaw crusher. The crusher product passes over a 5x10-ft mechanically vibrated screen (1½-in. opening), the oversize going to a No. 5½-standard cone crusher. The combined screen undersize and cone crusher discharge to a No. 5½-short head crusher preceded by two 5x10 ft mechanically vibrated screens in parallel (¼-in. opening). The materials are transported by belt conveyors.

The several materials handled are crushed to various sizes. By means of suitable by-pass chutes the materials may be drawn from the crushing circuit at the desired size. The limerock is crushed to $-1\frac{1}{2}$ in. $+$ ¼ in. for burning and screened to $-\frac{1}{4}$ in. for fluxing of concentrate. Mine ore containing a good percentage of free quartz is used for converter flux. It is crushed to $-1\frac{1}{2}$ in. The smelter reverts are crushed to $-\frac{1}{4}$ in.

Miscellaneous smelter materials, not requiring crushing, are brought in through the track bin and diverted ahead of the jaw crusher to by-pass the crushing circuit.

The crushed converter flux, reverts, and limerock for fluxing are stored separately in compartments of a 5200-ton storage bin. From there the materials are delivered to their destination by belt conveyors.

Bedding Plant

From the filter plant the concentrate is transported to the beds by a belt conveyor system, including traveling trippers and an automatic weighing machine. If desired, the concentrate may be sent directly to the reverberatory feed bins, by-passing the beds. A tripper spreads the concentrate, flux,

and reverts in layers over the length of the bed. Weights of all materials going to the beds are obtained by weighing machines. This permits accurate fluxing and charge calculations.

There are three beds 50x360 ft, each having a capacity of 11,000 tons.

After a bed is completed and properly fluxed a reclaiming machine excavates the charge for the reverberatory furnaces. There are two reclaimers and one transfer car by which the machines are moved between beds and to the repair bay. Each reclaimer has a capacity of approximately 200 tons per hr.

The reclaimer scrapes the material from the end of the pile and deposits it by means of a drag bucket conveyor on to a belt conveyor in a trench alongside of each bed. The machine has a reciprocating harrow set at an incline a little steeper than the angle of repose of the concentrate. As the reclaimer advances down the length of the pile the harrow removes a uniform cross-section of the layers of the concentrate, flux, and reverts. This provides a uniform reverberatory feed from each bedded charge.

A belt conveyor system transports the furnace charge to the reverberatory plant. The charge is weighed by an automatic weighing machine.

Lime Burning and Hydrating Plant

The lime burning and hydrating plant is operated to supply milk of lime for the concentrator.

Crushed limerock, $-1\frac{1}{2}$ in. $+\frac{1}{4}$ in., is stored in a 940-ton bin from which it is conveyed to a 50-ton surge bin ahead of the lime kiln. The kiln, 7 ft 6 in. x 315 ft long, is fed by a weighing feeder. It is fired with Bunker C fuel oil and has a rated capacity of 125 tons of burnt lime per 24 hr.

A ball mill and classifier are used to hydrate the lime. Insoluble cores produced as sands from the classifier are discarded. The milk of lime is pumped to the concentrator.

Reverberatory Plant

A traveling tripper, operating in a conveyor gallery above the firing ends of the reverberatory furnaces, delivers raw concentrate mixed with fluxing materials to charge bins, each having a capacity of 350 tons. Two such bins together with two smaller bins for silica or concentrate are provided over each reverberatory furnace. The small bins, each with a capacity of 100 tons, are used for fettling at the firing end of the furnace when converter slag is being returned. The traveling tripper also fills these smaller bins. Pan feeders, 54 in. wide, transfer the

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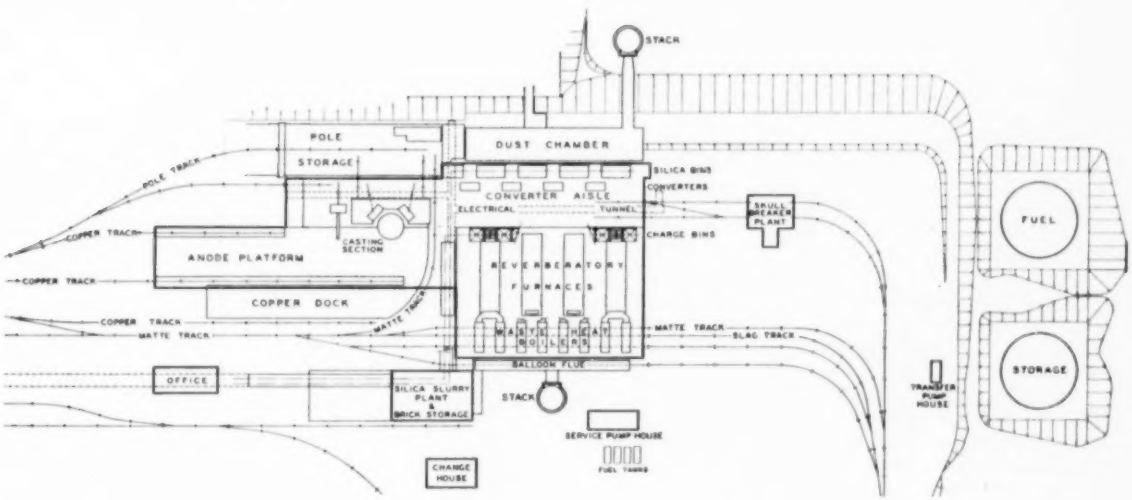
Smelter Plan and Equipment:



SMELTER UNITS

BEDDING PLANT

Three beds 50x360 ft, 11,000 tons capacity each

REVERBERATORY FURNACES

Four 30x125 ft furnaces, 650 tons of charge per day per furnace
Balloon flue and 300-ft stack

WASTE HEAT RECOVERY

Eight waste heat boilers
Four turbo-generators of 7500 kw each operating at 725°F and 400 psig

CONVERTERS

Four Peirce-Smith 13x30 ft converters
Three hundred-ft stack

CASTING WHEEL

Two casting furnaces 13x25 ft, 175 tons capacity each
Casting wheel with 26 anode molds in either of two sizes or 52 blister cake molds

STRAIGHTLINE CASTING

One holding furnace, tilting type
Endless chain casting section

One of the four 30x125-ft reverberatory furnaces before bottom pouring, shows burner-endwall bracing.

charge from the large bins to drag chain conveyors located over the furnace arches near the side walls of the furnaces. A pan feeder together with an adjoining drag chain conveyor operates as a unit and the drives of each are interlocked electrically so that neither one may operate without the other. The pair of pan feeders and drag chain conveyors over any reverberatory furnace may be operated simultaneously or separately to suit the smelting requirements. The charging rate is adjustable and is determined by the depth of the material being carried on a pan feeder. Fettling pipes, equipped with slide gates, direct the charge from the drag chain conveyors to fettling holes located in the furnace arch and spaced on 3 ft 8 in. centers to about 60 ft out from the firing-end wall. The slide gates are opened or closed manually from walkways provided along each side of the reverberatory furnaces.

The reverberatory furnaces are of similar design to furnaces now operating in the International smelter at Miami, Ariz., and were designed there under the direction of P.D.I. Honeyman, general manager of that plant. The furnaces are 30 ft wide x 125 ft long at the outside brick lines and will smelt about 650 tons of charge each per day. They are spaced on 64 ft centers. The roof arch is of silica brick, 20 in. thick, horizontal for about 58 ft and sloping from there to the throat at the uptake end. The side walls are of silica brick lined with basic (chrome magnesite) brick in the bath zone and around the tapping and skimming ports. The side wall thickness varies from a minimum of 1 ft at the top to nearly 5 ft at the bottom. The roof arch and side walls are insulated. The bottom of the furnace is of poured slag with a minimum thickness of 8 ft at the ends and 4 ft in the intermediate zone. The top of the slag bottom is at the same elevation as the floor of the reverberatory plant. A heavy concrete foundation retains the slag bottom, supports the furnace walls and buckstays, and takes the bottom thrust of these latter members. Pilasters, 18 in. wide, hold the buckstays out from the face of the furnace walls. The buckstays are on 5 ft 6 in. centers taking horizontal tie rods in the conventional manner. A longitudinal tie rod extends along the full length of each side of the furnace.

Converter slag is returned to the reverberatory furnaces in large matte ladles, each holding nearly 50 tons, and poured into a launder which enters the furnace through the center of the firing-end wall.

The bath depth is 3 ft. Matte is tapped through either of 2 tap holes in the flue-end wall of the furnace into short launders to fill matte ladles on special low-deck cars. Slag is skimmed through either side wall of the furnace, near the flue end, into longer launders to fill slag ladles in tilting cars. A ventilation system is installed over the tapping and skimming ports to remove the fumes.

Emergency tap holes are provided in each side wall of the furnace for use in the event of a break in the furnace wall. Trenches in the floor close by carry the molten metal under the matte and slag tracks to pool areas away from the smelter where it may be reclaimed when repairs have been made.

Each reverberatory furnace is fired by 12 oil burners mounted in groups of 6 on each side of the converter slag return launder. Bunker C oil is used. Blowers mounted on pedestals on the converter-aisle floor in a recess in front of the furnace foundations provide 25 pct of the required combustion air at 40 oz pressure. The remainder of the combustion air is drawn into the furnace through the burner ports and through pipe sleeves inserted in the upper part of the burner wall on each side of the converter slag return launder. Emergency burners, one to each side of a furnace, are provided along with separate facilities for furnishing combustion air required by them.

Exhaust gases leave each reverberatory furnace through a single 5x20½-ft outlet into an uptake which divides the gases for passage through the two waste-heat boilers provided for each furnace. The sloping floors and some parts of the side walls of the uptakes are self supporting but the roof and the balance of the side walls are of suspended construction. A portable, solid-brick damper carried on a trolley may be dropped into a slot in the uptake cross-over to cut out a waste heat boiler for repair.

Oxygen for lancing is available from a header with convenient outlets at each of the reverberatory furnaces. A central station, located in the silica slurry plant, provides facilities for putting 8 bottles of oxygen into service for lancing as well as storage space for additional bottles.

Clay for launder linings and silica for hot patching are ground and prepared in the silica slurry plant. The silica is pumped to agitator tanks located between each 2 reverberatory furnaces. A pipe loop around each furnace equipped with convenient outlets provides patching facilities to all parts of the furnace. Clay is delivered to the plant by battery powered lift trucks.

Slag ladles are of 225 cu ft capacity and the tilting ladle cars are drawn by battery and electric locomotives. Battery power is used within the smelter building and overhead trolley wires provide power beyond. The ladle tilting mechanism is controlled from the locomotive cab.

Converter Plant

The four Peirce Smith converters are each 13x30 ft, spaced on 64 ft centers which are staggered with the reverberatory furnace centers. The center of the converter is 17 ft above the converter aisle floor to permit the use of bulldozers for clean-up. The converter shells are of all-welded construction with the riding rings mounted on the ends. The converter lining is chrome magnesite (Kromag) brick throughout; the end lining is 27 in. thick and the shell lining is 18 in. thick at the bottom and 13 in. thick at the

top. The thickened end linings permit the use of only 50 tuyeres although the shells were arranged for 52. There are twenty 2-in. tuyeres and thirty 1½-in. tuyeres. All have special ball valves with renewable seats and are of non-clogging construction. The swivel joint in the blast line is constructed to eliminate external thrust. The quick-acting blast gate is operated by an air cylinder for blast-charge control. The puncher's platform is counterbalanced and is suspended by cables from above providing clear space below in either the high or low position.

The converter uptake hood is a special feature of the plant. It was newly designed to eliminate pockets where accretions might build up. The back wall slopes steeply so that the hood should be self cleaning. Copper shot or dust falling back within the hood returns into the mouth of the converter. A large counterbalanced gate opens high enough to permit pouring from the matte ladles or the pulling of collars when that becomes necessary. A jug-type damper and housing is installed on top of each converter uptake to insure the free opening or closing of the damper as well as for turning the converter gases into the brick dust chamber. The dampers are counterbalanced.

Silica for the converters is delivered to storage bins, each of 750 tons capacity, by a traveling tripper in the conveyor gallery above the bins which are behind the converters. The halves of 2 storage bins provide silica for each converter. Silica is fed to the converters through the side walls of the uptake hoods by means of small measuring bins which are in turn filled from the large storage bins. This permits the rapid charging of silica to either side of a converter overcoming the slowness formerly encountered when using Garr guns. Gates on the large storage as well as on the small measuring bins are operated by air cylinders controlled from the converter operating platform or cab. Facilities are provided so that Garr guns may be installed in the future if so desired by the operating staff. Provision has been made so that silica may also be loaded into boats for delivery by crane to the converter mouths.

Controls for the converters have been installed both on operating platforms between each converter and in individual cabs located on the reverberatory plant floor directly across the aisle from each converter. The cabs provide unbroken vision for the converter operations and are air cooled for the operator's comfort.

One skimming platform is provided for each converter and is of heavy concrete construction, the exposed surfaces of which are armored with steel grids. The converter operating platforms are of steel construction since they are not in the area where the battering by equipment in transit is to be expected.

Facilities have been provided for turning the converters and casting furnaces into position for emptying in case of a prolonged power failure.

Reverberatory furnace matte in 325-cu ft ladles is trammed into the converter aisle for delivery to the converters by overhead cranes. The ladles are equipped with 2 pouring spouts to eliminate the need for turning them when suspended from a crane hook. Special bails are provided for the large ladles. The arrangement of the converter skimming platforms affords more clearance for these ladles.

The converter aisle is served by two overhead cranes each with a 100-ton main hook and two 25-ton auxiliary hooks.

Converters are spaced on 64-ft centers, and staggered with the reverberatory centers on the main aisle.

Skull Breaking

A separate building, near the smelter is equipped with a 50-ton overhead crane and a hammer bridge for the removal of skulls from the ladles and the breaking of the skulls for return to the smelter. Ladles with skulls will be transported from the converter aisle on cars to the skull-breaking plant. The overhead crane will handle the dumping of the ladles to loosen and remove the skulls into a hopper with a grizzly bottom. The hammer bridge rides back and forth over the hopper so that an air hammer may crack the skulls to sizes small enough to pass through the grizzly openings. Gondola cars or flat cars with boats will receive the broken material for return to either the smelter crushing plant or the converters.

Casting Section

Casting of anodes and blister cakes will be done in a section west of the matte track, within the smelter, where two casting furnaces are placed to suit the mold spacing and unloading positions of the casting wheel and one holding furnace is placed above the straightline casting machine. The 13x25 ft furnaces are of the tilting type, and may be completely rotated for emptying into the pouring spoons. They are of similar design to the converters and the riding rings, ring gear, pinion gear, and drives are interchangeable with those of the converters. The furnaces are lined throughout with chrome magnesite (Kromag) brick backed with insulating brick to reduce heat loss. The bottom lining is 18 in. thick and the top lining is 13 in. thick. Each furnace is provided with an air atomizing oil burner in a hinged mount. Combustion air for these burners is supplied by 2 sets of blowers conveniently located near the casting furnaces. Separate air-cooled operating cabs are provided for each furnace. A poling platform 38x120 ft, of heavy concrete construction with armored surfaces, is provided around the two anode casting furnaces at the casting wheel.

Matte from the converters is delivered to the furnaces, which hold about 175 tons each, in the large matte ladles by the converter-aisle cranes. Anodes or blister cakes are unloaded on the anode platform area south of the converter aisle. The two anode casting furnaces at the wheel are equipped with extended charging spouts and poling ports and

Looking down at the smelter, power plant in foreground, converter stack at left, and reverberatory stack, at 66 ft lower elevation, at right.

the holding furnace is equipped with a 6x6-ft charging door; burner ports and gas outlets are provided in all of the casting furnaces. Small ladles (each of 125 cu ft capacity) placed on self-propelled cars with remote control, are used for the removal of slag from the casting furnaces. Tracks for these cars extend from the north side of the converter aisle to positions which permit the spotting of a ladle beneath each furnace. Operation of the cars is controlled from panels on the columns across the converter aisle from the casting furnaces.

A battery-operated crane truck on the poling platform of each casting furnace at the wheel transfers poles from special bin cars in the converter aisle to the poling platform or inserts the end of a pole into the poling port on the furnace. A frame, over each of these furnaces, with rigging operated from an air hoist is employed to handle the poles after they are placed in the furnaces. Charging racks on the poling platform assure maintenance of the crane truck batteries. Space has been made available north of the converter aisle for the storage of 6 months' supply of poles.

A movable platform, remote controlled, has been installed on rails to furnish access from the poling platform to the pouring spouts of the casting furnaces and to the deck of the casting wheel. When not in use this platform is spotted over the casting wheel near the columns south of the casting furnaces.

The casting wheel is of new design and is arranged to handle 26 anode molds in either of two sizes or 52 blister-cake molds. It has a flat deck giving greater working area around the molds. Stationary rollers support the wheel near the rim and a roller bearing is placed under the center. The driving motors are placed in a pit below the wheel and the drive pinions engage an internal gear bolted to the bottom surface of the wheel. Acceleration and deceleration of the wheel has a special feature of control incorporating a pilot flywheel. Operation of the wheel is so smooth that the wheel may be moved with no delay for cooling. The drive is reversible permitting the wheel to turn in either direction for the long or short travel of anodes from either casting furnace to the unloading position. A system of sprays above and below the molds has been provided for cooling. Operation of the wheel is controlled from either of the two cabs provided for the casting furnaces.

Copper is poured into the molds on the wheel from pouring spoons, hydraulicly raised or lowered, under the pouring spouts of the casting furnaces. The pouring spoons are of steel plate and lined with brick and a mud mixture. A built-in skimming bar holds back charcoal when copper is being poured. The tilting and levelling of the pouring spoons is controlled from the operating cab at each furnace.

Hydraulic rams raise the knockout pins in the mold when the mold reaches the unloading position. The wheel is locked in the unloading position while the anode or blister cake is being lifted out of the mold and may only start its travel again after the ram has been completely lowered. Bosh tanks are placed opposite the two unloading positions for anodes and two other unloading positions are on either side of these for the unloading of blister cakes. Controls for the rams are placed conveniently for access by the operator.

An overhead crane operating in an arc over the four unloading positions of the casting wheel is equipped with an air-cylinder-operated trolley from which an air-cylinder-operated hoist is suspended to remove the anodes from the molds and place them in either bosh tank or to lift the blister cakes from the molds to place them on pallets. The racks in each bosh tank will hold 38 anodes and, when filled, a rack is lifted from the tank by a 15-ton overhead crane and placed on the anode platform. Battery powered fork-lift trucks handle the anodes through weighing, tilting, and placing in storage on the copper docks. An electrically operated tilting device turns five anodes at a time from the vertical to a horizontal position for bundling prior to shipment in gondola cars.

The straightline casting machine is of conventional design, being an endless chain equipped with molds for the continuous casting of blister cakes. Control of the straightline machine, the holding furnace, and the pouring spoon is operated from the cab. Battery powered fork-lift trucks handle the cakes after they are dropped from the molds.

Most of the finished product from the plant will be loaded on cars inside of the casting section where two copper tracks have been located. Copper dock space has also been provided outside of the smelter building.

Handling of Flue Dust

Dust which is collected in the reverberatory balloon flue will be transferred by a drag chain conveyor to a bin, thence to a tank from which it is blown to the silica slurry building where it is pugged and returned to the furnaces with the charge on the conveyor.

Dust from the converters will be collected in the bin bottoms of the brick-dust chamber by drag chain conveyors into a cross drag chain conveyor to a bucket elevator serving a bin in the converter aisle. Boats are filled from this bin and are then transported by crane to return the flue dust as cold dope to the converters.

Smelter Stacks

Smelter stacks, in addition to withstanding earthquakes, had to provide for operations under a wide variety of conditions. Although one reverberatory furnace and two converters might be operated initially, the completed plant could have five or six

furnaces and eight or ten converters running simultaneously. A number of small stacks was decided upon rather than a single tall stack.

The smelter has been located so that the prevailing winds will carry fumes away from the remainder of the plant. A height of 300 ft was selected for the stacks, with the top of 24 ft 9 in. diam clear inside of the lining. The converter stack is located on a hill alongside the smelter, raising it 66 ft above the reverberatory stack.

Steel was chosen in preference to reinforced concrete for the main part of the stacks because of the lightness of a steel structure and the ductility of the metal itself.

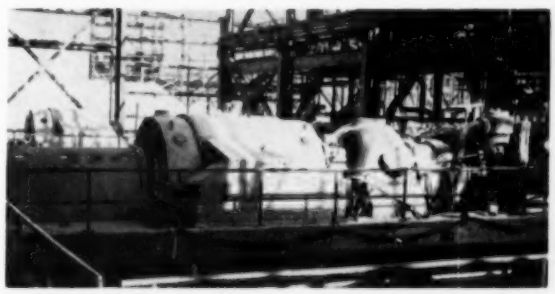
The main shaft of the stack is steel. The shape is a frustum of a cone tapering from an inside shell diameter of 25 ft 7½ in. at the top to 35 ft at the bearing plates. The lining of acid-proof brick is laid in Sauereisen cement mortar, with about ⅜ in. of this mortar between the brick and the steel shell. The lining is 4½ in. for the top third, 9 in. for the middle third, and 13½ in. for the remainder. The top is protected by an annular ring of replaceable cast iron segments.

The converter stack will have only the bottom portion of the lining installed at first. This part is needed to protect the concrete from heat and possible condensation of acid.

Waste Heat Power Plant

COPPER reverberatories develop large amounts of exit gases of a temperature in the neighborhood of 2000°F. The gases are not only of a noxious nature but must usually be disposed of at considerable heights above the yard level by a stack. Handling these gases at the temperature mentioned would require rather expensive construction of conveying flues and stack. Moreover, wasting such amounts of heat is certainly most uneconomical.

Numerous investigations have indicated that waste heat boilers of the water tube type are the most practical and economical means for utilizing that heat although the gases are heavily laden with rock dust and copper-bearing slag, which upon cooling form hard deposits on the surfaces of dampers or heat absorbing elements placed in the gas stream.



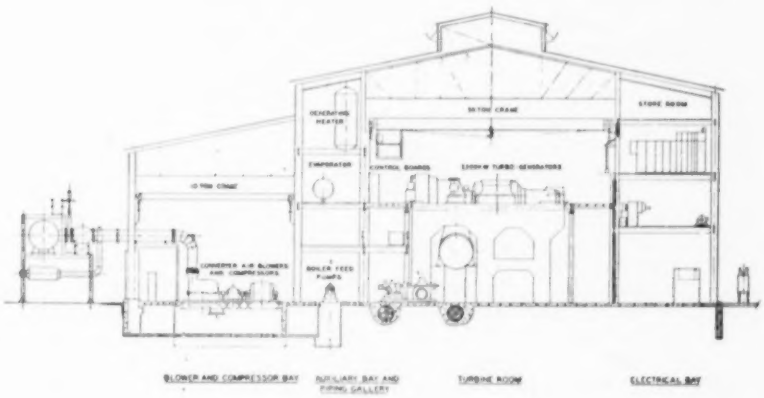
Four 7500 kw turbogenerators are installed. Capacity was chosen to absorb at best load amount of steam generated by one reverberatory furnace, in line with the arrangement of concentrating and smelting equipment on the unit system.

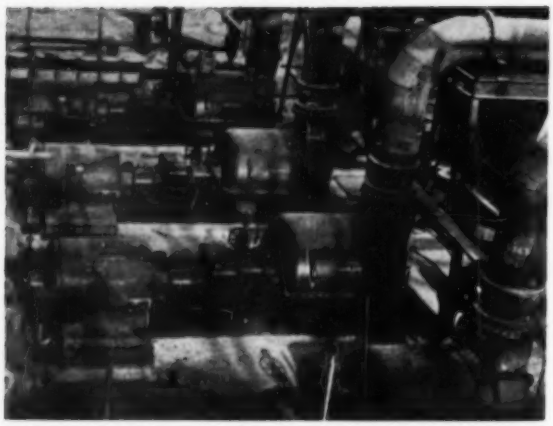
Avoiding Slag and Dust Deposits

Based largely on the experience of the paper industry in burning black liquor, a waste-heat boiler design was develcped a few years ago, providing not only extensive water-wall surfaces for cooling the gases below the fusing point of the slag but also settling out, as far as possible, the solidified particles of slag by creating low gas velocity. Matter carried in a stream of gases or air has a tendency to maintain its original direction upon a change in the direction of the flow of the conveying medium. Reverberatory waste heat boilers installed at Hurley and Morenci endeavored to utilize that tendency for the elimination of slagging particles at predetermined places and before they would strike the tube banks of the boilers.

In line with Anaconda practice each reverberatory furnace is equipped with two waste heat boilers independent of each other, so that one may be cut out in case of tube failure without necessitating the shut-down of the reverberatory furnace. The waste heat from the reverberatory furnaces is sufficient to

Waste heat steam is supplied at a temperature of 725°F to the throttles of the turbogenerators in the power plant. The waste heat power plant also houses motor-driven compressors for converter air supply.





Converter air is supplied by these four motor-driven turbo-blowers of 30,000 cfm capacity each. Blowers are direct connected through speed-increasing gearing to induction motors, and operate at constant speed and pressure.

operate a steam power plant having the capacity to generate power for 40 to 50 pct of the requirements of the concentrator, mine and crushing plant.

Power Generation

The purpose of the waste heat power plant is not only to provide an outlet for the steam generated in waste heat boilers but also to form a source of emergency power in case of loss of supply from the Tocopilla power plant due to strike, sabotage or disaster. Furthermore, provisions have been made for auxiliary oil burners in the front walls of the boiler furnaces to permit their use as direct-fired boilers for furnishing steam to generate power entirely independent to Tocopilla supply and after a complete shutdown of the sulphide plant.

In view of the extreme slagging tendency of the gases handled, a superheating section is an undesirable feature in copper reverberatory waste heat boilers on account of the close spacing of its elements. Except when used in combination with direct-fired boilers, as at Morenci, separately fired superheaters are uneconomical and could not be justified for the Chuquicamata plant. The temperature of the waste heat steam was therefore arbitrarily limited to a maximum of 750°F, resulting in a temperature of 725°F at the throttles of the turbo-generators.

Although higher steam pressures were investigated, a throttle steam pressure of 400 psig appeared to offer best returns on investment and was adopted. Furthermore, since the amount and temperature of the waste heat steam coming from copper reverberatory plants is dependent upon the charge and fuel ratio of the reverberatories and beyond the control of the power plant operators, it was considered advisable to provide automatic temperature control by installation of de-superheating equipment in the steam line to the power plant.

The Oxide Plant

CHILE EXPLORATION CO.'s oxide plant at Chuquicamata, the largest of its kind, started in 1915 with initial operations at the rate of 10,000 tons of ore per day. Anaconda Copper Mining Co. acquired the controlling interest in 1923 and in the next few years modernized the plant and mine, extending operations to a nominal capacity of 490 million lb of electrolytic copper yearly by 1942. Plant production averaging 495.7 million lb of copper per year was maintained for the eight-year period extending from 1941 to 1948 inclusive. During World War II production reached its highest level when in 1944 the output was 531.8 million lb. The maximum production for any one month was 49.6 million lb, achieved in May 1947.

The oxide plant is divided into five main operations as follows: 1—Crushing, 2—leaching, including dechloridizing and sulphur dioxide treatment, 3—tailing disposal, 4—electrolytic precipitation, 5—smelting and melting.

Crushing

The ore as received from the mine is frequently blocky and up to 5 ft in section. It is reduced to 9-in. size by two 60 in. gyratory crushers, and transported to the ore bins by 60-in. conveyors.

In the secondary crushing operation the ore is first reduced to 2 to 3-in. size by five standard 7-ft cone crushers and two No. 10 gyratories. This product is then reduced to $\frac{3}{8}$ in. size by fifty-two 48-in. vertical Symons disk crushers and is transported by belt conveyors to the loading bridges from which it is charged into the leaching vats.

Leaching

The ore is leached in 14 vats, averaging 13,500 short tons net capacity each. These vats are lined with a natural mastic which adequately resists the sulphuric acid leaching solutions.

The leaching process as presently conducted comprises soaking the ore with successive volumes of leaching solution and spent electrolyte and withdrawing successive volumes of enriched solution from the vat to a total of approximately 3.2 million gal. The first 1.6 million gal of this volume withdrawn is called strong solution and contains about 35 parts of copper per 1000. The balance of 1.6 million gal subsequently withdrawn is stored in a solution sump and later withdrawn therefrom to cover a succeeding vat of fresh ore and to produce strong solution from it. When this intermediate solution is withdrawn from the ore, fresh spent electrolyte and solution advanced from the washes replace it. The ore following these soaking or leaching periods is then washed with a series of five wash solutions each containing decreasing concentrations of copper and sulphuric acid and is finally washed with a volume of 396,000 gal of fresh water.

Approximately 264,000 gal of moisture are absorbed by the unusually dry ore which contains about 1½ pct moisture as mined, and about 26,400 gal are lost in evaporation. The difference of 106,000 gal of fresh water is therefore advanced through the washes and into the primary leaching solution for each vat of ore treated, and a corresponding volume of 106,000 gal per vat is stripped of its copper and discarded to waste thereby effecting control of impurities, particularly iron, molybdenum, arsenic, etc. To further increase the efficiency of the leaching process an additional 106,000 gal of wash water per vat is passed through special electrolytic cells wherein a portion of its copper is removed and the solution returned to the washing system.

The method of leaching employed is a batch system and the total cycle time from the start of loading the ore into the vat to the termination of the excavation of the leached tailings averages about 72 hr. The leaching plant, therefore, has a capacity of approximately 4.7 vats per day.

The mineral antlerite, a basic copper sulphate, supplies the bulk of the sulphuric acid gained from the ore. This acid gain is currently 3.3 to 4.4 lb of sulphuric acid per metric ton of ore. To augment the amount of acid required to operate the leaching system sulphur dioxide is added to the strong solutions just prior to electrolysis, in amounts ranging from the equivalent of 0.44 to 3.1 lb of sulphuric acid per ton of ore leached. As the electrolyte contains nitric acid, chlorides, ferrous iron and molybdenum compounds leached from the ore, the sulphur dioxide added to the solution before electrolysis serves additionally to stabilize it by preventing decomposition of the nitric acid and self-oxidation of the electrolyte.

Recovery in leaching amounts to about 95 pct of the oxide content of the ore and 10 pct of the sulphide content. The average overall recovery on the 360 million tons of ore treated in the plant from the start to Dec. 31, 1951, which contained 1.70 pct total copper, has been 83.75 pct. Though the nominal rating of the plant is 1.4 million short tons of ore per month, a total of 1,666,748 tons were treated in June 1952.

Chlorides, which are present in the ore and also in the water used for washing, are precipitated as cuprous chloride by agitating the strong solution with finely divided cement copper. The cuprous chloride is subsequently settled out of the solution and recovered. This process takes place before the solution is delivered to the electrolytic cells. The cement copper is regenerated by dissolving the settled out cuprous chloride in a ferrous chloride brine solution which is then passed over scrap iron in 7-ft diam bronze drums.

Electrolytic Precipitation

The tank house building, 1187 ft long x 160 ft wide, is divided into three bays. There are 1098 mastic lined tanks in the building, each approximately 19x4x5 ft deep.

In the commercial cathode sections, 67,556 lead-antimony-silver anodes equipped with long mastic strip insulators are in service, and 5457 anodes of a similar type are employed in the commercial starting sheet sections. In general, 16 or 17 tanks comprise a section and eight sections in series electrically make a circuit, of which there are nine in the house. Each circuit is powered from one or more rotary converters or motor generator sets. Current density averages 14 amp per sq ft. Anodes are placed on 3-in. centers. Magnetite, ferrosilicon and copper silicide (Chilex) anodes have all been used in the past, the latter having been replaced entirely by the present lead-antimony-silver anode.

Efficiencies average 85 to 92 pct with a yield of 25 to 28 lb of copper deposited per kw-day under normal conditions. Commercial starting sheets deposited from strong solution with insoluble lead-antimony-silver anodes are used.

The inflow solution to commercial cells is made up of starting sheet return and first stage spent electrolyte, part of which has been passed over the towers of the sulphur dioxide plant; and second stage-spent electrolyte averaging 15 parts of copper per 1000 is returned to the leaching plant. Flow rates of solutions vary from 200 to 265 gpm. Temperature of the electrolyte is held between 36° and 30°C.

Neoprene sleeves are used on all anode tail bars and the development and use of the long mastic strip insulators has eliminated at least 50 pct of the inspection required in former years. Cathodes deposited to 150 lb are transported to the furnace dept., where market shapes are cast.

Furnace Dept.

Fourteen different electrolytic copper shapes are commonly cast by the furnace dept. including 10 sizes of wire bar varying from 135 to 310 lb, end poured wedge cakes, flat cakes, billets and ingot bars.

Three reverberatory-type market furnaces of 400 tons daily capacity having a hearth length of 49 ft 5 in., and width of 13 ft 10 in. are in service. The bottom and roof are of silica brick, and the side walls are magnesite lined. They are charged through three charging doors by Morgan open hearth charging machines.

The charge consisting of hard dense pure cathodes plus rejected shapes, warmers or skulls, is expertly packed into the furnace by two charging cranes, after which the furnace is sealed and the melting operation started. Fuel oil burned by two burners is gradually brought up to 460 gal per hr, and held at that rate until the end of the melting down period.

Boiling caused by the melting of cold metallics on the bottom of the furnace, takes place within 8 to 10 hr, after which the fuel oil is decreased and the charge skimmed clean. Air at 12 lb pressure is blown into the molten metal by iron pipes inserted through small openings in the door. This is continued until the granular texture of the test buttons indicates that approximately 0.8 pct oxygen is contained in the metal and that scorification is complete. The oxidized-silicate slag formed largely by erosion of roof brick and bottom is then skimmed off. After skimming the charge is covered with charcoal and the reduction of copper oxide by means of poles forced into the metal bath is started. When the oxygen has been reduced to 0.03 to 0.04 pct the furnace is tapped and casting starts. Each furnace is equipped with a 40-ft diam Clark-type mechanical casting wheel. All molds are made of refined copper and are replaced after four to seven charges. The cast bars or shapes are given a rigid inspection by two inspection crews and rejects are remelted. After the second inspection, the bars are weighed on the production bullion scales and prior to shipment are lot marked and re-weighed on the shipment scales.

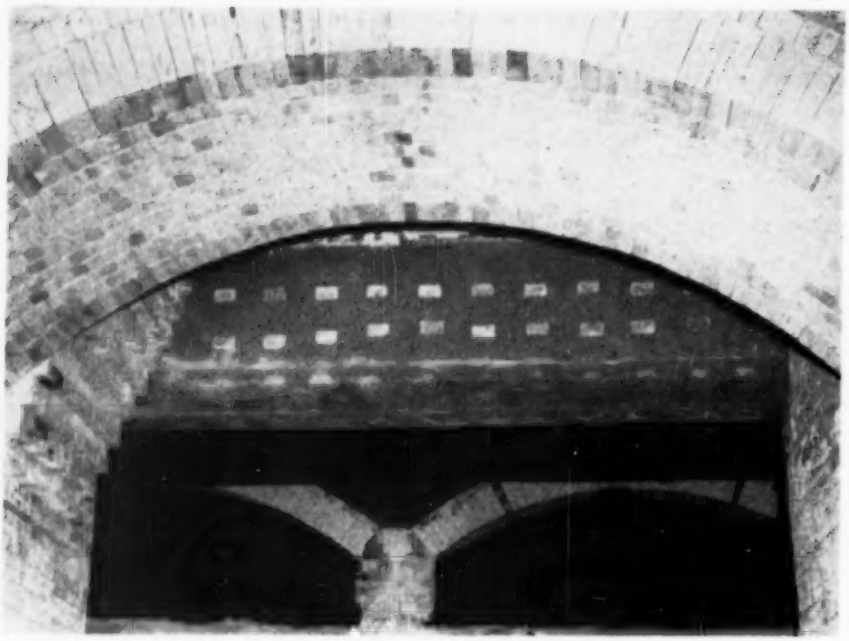


Fig. 1—This view of a rammed fantail on an open hearth furnace was taken from the pit side of the slag pocket.

Rammed and Castable Refractories Find Increased Steel Plant Use

by R. Russell Fayles

ONE of the outstanding trends in the refractory field has been the rapid growth in the use of rammed and castable materials for steel plant service. Possessing high temperature capabilities, adequate strength, and good expansion characteristics, they have found increasing favor throughout the industry. Such materials have been available and in use for some years, but within the last five years usage has increased tremendously.

A review of plant practices at Lukens Steel Co. with these products probably will establish a pattern similar to that of other companies, varying only in degree. The first use of plastics came in the late 1930's with the adoption of plastic chrome ore for open hearth door linings. The old conventional clay brick door lining with its 10 heat life was converted to the studded, rammed, and steam dried plastic chrome ore lining which now gives upwards of 110 heats per lining. Experiments with various door lining methods and materials have been many and varied, but up to this time, plastic chrome ore has rendered the superior service. Chrome castables presently are being tried for this service, but insufficient factual data is available to establish conclusions.

The use of rammable materials in bottom construction also began in the late 1930's, and included both plastic chrome ore and magnesia materials for subhearth areas. Both types of refractories have been used for such service on all new open hearth bottoms installed since 1940. Gradually, the use of this general type of material has been extended until now, high magnesia, rammed refractories are used for extensive areas of the hearth. Gone are the days of lengthy bottom sintering prevalent when magnesite was the preferred bottom material. Valuable time has been saved and additional steel produced by the rapid bottom-making possible when using rammed hearth materials. While no definite data is available, the large number of rammed open hearth bottoms already installed or contemplated is indisputable evidence of the acceptance of rammed refractories for this important furnace service.

The miscellaneous uses for rammed or castable refractories in open hearth service constitute an interesting although not too important item. Because of varying shop factors, such uses are generally spotty and cannot be copied except with caution. Some of these uses are special linings for tapping spouts, facings for bridgewalls, burner port coverings, and others. It is always interesting to learn of the many refractory trials made for such purposes and to speculate on the chances for successful application in one's own plant. In this miscellaneous category could be included gun emplaced refractories used for various points of furnace maintenance. This operation has enjoyed varying degrees

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of success, depending on shop conditions. Theoretically, the operation offers definite value to any plant. However, a combination of indifferent refractory performance, charging floor nuisance, and furnace down time have limited its use. The refractory gun presently is being used for spot repairs to prolong furnace life or postpone repairs, and also to spray regenerator roofs to reduce air infiltration. Experiments are being conducted with gunned refractories made at Lukens in an effort to improve both the shooting operation and the performance of the refractory.

Most of the foregoing has been a prelude to the major campaign presently under way. Systematically throughout the entire plant, sprung arch or suspended brick constructions are being eliminated wherever possible in favor of suspended rammed or castable refractories. In addition, the possibilities of substituting these materials for soaking pit walls and other areas involving firebrick construction are being probed slowly. This plan has been motivated by a single purpose to reduce masonry man-hour requirements to the lowest possible level. Bound down by a bricklayer shortage and an inability to attract apprentices, plans are being directed toward the end that masonry crews can utilize nearly all their efforts towards rebuilding open hearth furnaces. In conjunction with this plan, the thesis was developed that unskilled labor can install satisfactorily these special refractories. At the present time, 24 soaking pit covers, 4 ingot heating furnace roofs, 7 reheating or heat treating furnace roofs, and 3 open hearth furnace fantail roofs have been constructed of rammed or castable refractories. The program is steadily being pushed forward, and it is now specified that all new miscellaneous heating furnaces must have plastic or castable roofs for the original installations. Slowly but surely, the point is approaching where almost all masonry hours can be devoted to open hearth work and appreciably speed up furnace rebuild time. The happy part of the entire program is that the new suspensions outlasted the old types from 2 to 5 times, and so reduced maintenance costs and outage times, showing a tidy cost saving in every case. Thus, the effect has been to please top management costwise, operating supervision by reducing delays, and to ease bricklayer shortage to a marked degree.

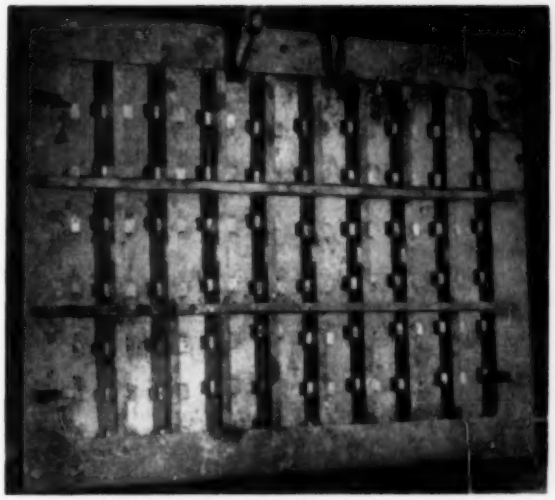

Fig. 2—The cold side of a castable cover for regenerative pits is shown.

Naturally, much interest is focused on experience with open hearth rammed fantail roof construction. This particular phase of the program was of secondary interest when the program began, but success in other parts of the plant prompted the taking of a calculated risk trial on a single open hearth furnace. This initial installation was made on a 135-ton furnace originally built for 75 tons, with two regenerator chambers at each end (producer gas fired originally; now oil fired). The division wall between the chambers extended to the slagpocket area and created rather restricted, crooked fantail openings into the checker areas. The roofs were entirely of sprung arch construction, using semi-silica brick throughout. The fantail roof zones were prone to air leakage and rather high maintenance costs. Extensive repair or complete replacement was usually necessary within 500 heats and an embarrassing furnace shutdown was sometimes required when the arch in the sidewall at the juncture of the wall and fantail collapsed. Redesign called for the elimination of the central division wall from the slagpocket area to a point 7 ft beyond the checker bridgewall. The

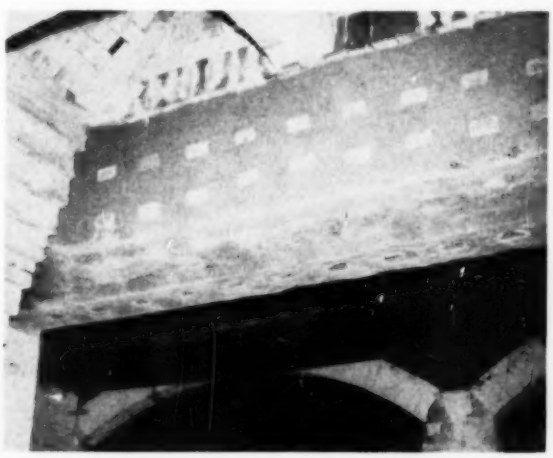

Fig. 3—This is a close up view of the open hearth rammed fantail shown in Fig. 1.

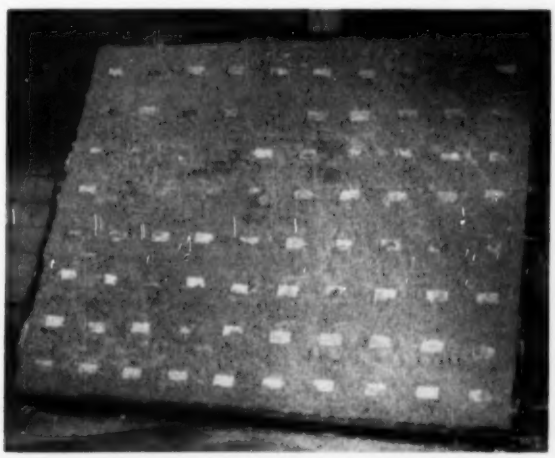

Fig. 4—The complete hot face of this rammed cover for regenerative pits can be seen.

entire fantail roof from this point to the nose at its junction with the vertical uptake wall and a short distance up the wall was of suspended, plastic firebrick construction. These changes gave an improved gas flow pattern into the checkers and a slight increase in checker volume because of utilization of the volume formerly occupied by the division wall. The monolithic, rammed roof construction provided a design that virtually eliminated air leakage, gave promise of extended service life and simplified repair when and if required. There was tremendous interest in the effect of these design changes on furnace operation as well as the performance of the rammed refractory. It is happily reported that the operating results surpassed fondest hopes. On the first redesigned furnace, the production rate increased 11 pct and the fuel rate decreased 10 pct, with all other factors as nearly constant as routine operation would permit. Spurred by this initial success, the design changes were duplicated on another furnace, which showed an 11 pct production increase and a 4 pct fuel decrease. Still another furnace was similarly changed, except that it was of basic end construction with a basic fantail nose and the rammed plastic fantail roofs. This unit only has 100 heats out on the run, but compared with a like period during the previous run shows a 9 pct production increase with a 4 pct fuel decrease. The first redesigned furnace now has made 480 heats since the installation of the rammed fantails and has had only a small repair to one roof, occasioned by an overload of dolomite and lime which piled up on the suspended section. These areas are now fully protected and further trouble from this source is not expected. Inspections have failed to reveal the slightest wear on the fantail or regenerator roof sections. They give every evidence of lasting indefinitely. There is an appreciable wearing action on the face of the nose occasioned by the flow of molten silica brick from the wall above. This sloughing action appears to cut away about 1 in. of the plastic on each roof run of 180 to 200 heats. This erosion only occurs on that portion of the nose directly under the uptake wall

Fig. 5—A close up view of rammed fantail after an open hearth furnace campaign.

and, when repairs do become necessary, they are minor. It should be noted that all three of these installations came through the recent strike shutdown without the slightest sign of deterioration aside from a couple of shrinkage cracks which have closed completely since the units are again in production.

Furnace redesign as described has been found to be completely satisfactory. The performance of the furnaces and refractories has been excellent in every respect. The installation costs have been on a par with, or less than, an equivalent suspended brick construction and, in every case, have been completely paid for from savings during the first roof campaign. No changes made in furnace design or steelmaking practice during the author's 11 years with the company have ever shown results like those possible with the changes described. As rapidly as rebuild schedules permit, all furnaces will undoubtedly be changed to the new design and incorporate rammed refractories in all installations.

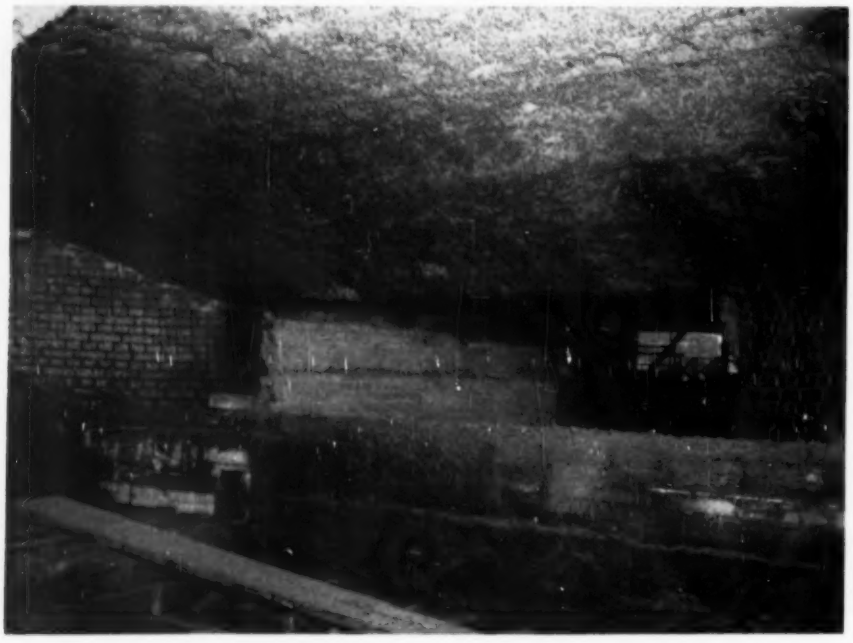

Fig. 6—Rammed fantail view after an open hearth furnace campaign, as seen from regenerator.

Select Engineer Employees Scientifically

by F. R. Morral

- Productivity with Higher Efficiency
- Need of Better Use of Human Resources
- Encouragement of Personal Pride in Work Performed
- Employee's Happiness on the Job and Away From It

INDUSTRY has yet to find a universal solution to the problem of engineer personnel selection. Today, the choice of the right man for the right job is even more pressing than ever before. The age of the hard apprenticeship seems to have passed. In its place one finds the interviewer who must determine through personal contact and evaluation of college grades whether a graduate engineer will fit into his company's scheme. The problem, as it is constituted today, may be broken into four parts:

Need for better use of human resources (there is a shortage of engineers, scientists, and skilled workers),[1] productivity with higher efficiency,[2] encouragement of personal pride in work performed, and employee's happiness on the job and away from it.

The last three items are somewhat related and the attendant results, unfortunately, make an imprint in everyday life. Products advertised as improvements and gains of the technological age do not come up to promised expectations. Citizens and industry complain about taxes and government expenditures, but soon turn to the government for special privileges and tariffs. Absenteeism and job turnover is at a high level. Crime, divorce, ulcers, and accidents are rampant. Is it possible that too many square pegs are being pushed into round holes?

College graduates in engineering and science are expected to grow into responsible positions in production, development, research, and sales. However, on graduation their potentialities are unknown. The colleges have given them *know how*, *know what*, insufficient *know why*,[3] and some *know where*.

Scientific methods seem to be necessary to place the graduates properly. This could be followed by training to help them develop to the maximum of their individual capacity. Eventually, they would step into the available responsible positions. This would help do away with any one, and possibly all, of the problems listed above.

Most companies will admit that employee selection practice is often no more than a *trial and error* proposition. It may be pertinent to ask: How does the college graduate and the company choose the course taken?

At an interview, both employer and employee are on their best behavior, wearing their Sunday suits and best of grooming, and on the watch for the proper choice of words. Grades, activities, hobbies and other factors are discussed. The interviewer points out the glamorous side of his company, benefits, pay, vacations, the presidency as a goal. If mutually acceptable, the graduate may get a special indoctrination course. Only a few years back it was customary for the graduate to learn the business by starting at the bottom of the ladder, using pick or shovel, or pushing a wheelbarrow.

Men in the mining and metallurgical industry, particularly in the foreign field, have been able to show that they *had what it takes* and in only a few years rose to responsible positions. In this country some stopped on the way and were satisfied with their place. Others considered themselves misplaced. They were disgruntled but thankful for a living. A few companies have been disturbed about this group and have done something. Some people do something themselves. They either ask for a transfer within the company, or just wait for the opportunity to try the greener grass on the other side of the fence.

All companies are conscious of need to use an individual's capacity to the maximum, since this is

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the key to successful operation, both from the point of view of the individual as well as that of the company. How successful a company is with their personnel can be discerned in their records on absenteeism, accidents, turnover, promotions and possibly demotions. These records also reflect the company's technique of employee selection' and choice of training and rating methods.[5] The training of science and engineering graduates costs more than that of other technical labor. In this country, as well as abroad,[6] some companies have been looking for scientific methods of selection of professional employees.[7]

The choice of such personnel is made by giving batteries of tests, some of which are used in schools. They may also be employed in the selection of less skilled personnel. Personality tests have not been as common because more research seems to be necessary to evaluate and use them. In summary, the tests given are:

► Aptitude tests, which have been long accepted by industry as valuable tools for personnel selection, training, and promotion[8, 9]

► General intelligence tests

► Interest type tests,[7] which measure personal and job interest—This is designed to show a candidate's interest in a few or all the following fields: Scientific, persuasive, mechanical, computative, clerical, social service, musical, artistic, and other more specialized activities.

► Personality tests which cover mental aptitudes, drive,[10] academic achievement, judgment, reasoning, dexterities and emotional adjustments, attitude, and imagination — The latter should be particularly important for the selection of research and development personnel.

Data obtained from the above tests must be evaluated and correlated with the characteristics or attitudes required by the position. Each job requires proper balance of fundamental attitudes for success. If these match, it may be possible to have a satisfied employee with pride in his job, company, and product. Such a program of psychological tests for graduate engineers might help the mining and metallurgical industry.

How could a better use be made of human resources? By the selection of human beings who have *what it takes* and would *like to do it*, and make it possible for them to earn the proper training. Considerable data is gathered on the human being while he goes through the various stages of life starting with grade school. However, this information is not correlated, and, therefore, goes to waste. It seems that this material could be used to everyone's benefit. For instance, many of our children are given intelligence tests while in grade school and later on. Some are given aptitude tests before they graduate from high school. It appears that there has been some reluctance in using this data to advise and to follow up. The purpose of data, it would seem, is not to use it as a club, but as an aid. At present those who can afford to go, and some with stamina who can not afford to, proceed to college. In many universities the first week as a freshman is devoted to taking batteries of tests that find their way to Dean's offices, where they often gather dust. If the student gets into difficulties the results are dug out, and a well meaning person may try to counsel the unhappy and unsuccessful student. In some engineering colleges, if the student does not make the proper math, physics, and chemistry grades, he is advised to change fields.

A few college graduates are not satisfied that they know their calling. So they go to a university or private psychological laboratory for further study and interview and seek advice and help.[11]

Some industrial companies[5, 12, 13] give a set of tests to job applicants. The tests vary according to the needs of the position.[7] The aim is to fit the round peg into a round hole, and to make the best use of the candidates' potentialities. It is hoped that in future discussions the experience of some companies will be made available.

In conclusion it appears that considerable data is collected, but ignored. Intelligence tests, aptitude tests, interest tests, grades, rating by teachers, psychologists, and professors are available, but only little of this material is correlated in a single continuous personal record. This record could point out the amount and the rate of improvement made by the person, and give information which could be used to permit society to choose and nurse the potentially valuable, but now lost human resources and put them to the best use. The individual would be advised of his shortcomings and encouraged to overcome them. The company would be better able to direct human beings toward openings where candidates would be happy with the environment and take pride in production. Psychological testing and evaluation programs need not be limited to large companies which have huge personnel departments and possibly their own psychologists.[6] It can be adopted by small companies, if they take advantage of consulting services offered by private and university psychological laboratories. The fee certainly will not be as high as the cost of training the graduate, and the accomplishments may result in repayment in a short time.

A word of caution is perhaps necessary. As in everything else, the services of reputable and competent psychologists should be sought. Further, tests reduce but do not eliminate mistakes in picking the right person for the right job.

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Domestic Chrome and Manganese Ores Can Be Upgraded and Utilized

by H. A. Doerner

METALLURGICAL problems relating to manganese and chromium ores have striking similarities. Ferroalloys, essential to the steel industry, are produced from both ores. Most of these alloys are obtained from imported ore, as domestic deposits have not yielded enough ore of metallurgical grade to meet more than a small fraction of vital needs.

A survey of domestic manganese and chrome deposits reveals numerous small, dispersed bodies of fairly good ore, none of which appears to be capable of yielding significant amounts of marketable products. To stimulate development of such deposits and to increase Government stockpiles, General Services Administration is offering $115 per long ton for limited amounts of chrome ores and $110 per long ton for 48 pct manganese ores that meet metallurgical specifications, with bonuses or penalties for grades above and below specifications.

Even at these prices, domestic output of manganese and chrome ores is far below metallurgical requirements, and other measures would be necessary to prevent disaster if imports should be cut off by enemy action. Since private industry has little incentive to develop methods for utilizing submarginal ores, this problem became a responsibility of the Bureau of Mines.

A few domestic manganese and chromite deposits are known to contain large bodies of ore that can be mined economically by non-selective methods. These include chrome deposits near Columbus, Stillwater County, Mont., and low grade manganese ores at Artillery Peak, Ariz. Unfortunately, these chrome and manganese ores are not only low grade but will not yield metallurgical-grade concentrates by any ore-dressing treatment.

Chemical methods usually are required to convert low grade manganese and chrome ores into products that meet the specifications based upon high grade imported ores and demanded by producers of ferroalloys. Numerous chemical methods have been investigated by the Bureau of Mines in persistent efforts to upgrade domestic manganese and chrome ores or to convert them to salable products, such as commercially pure oxides, salts, and electrolytic metals.

At its Boulder City station, the Bureau of Mines has had notable success in pilot-plant development of electrolytic processes by which high-purity manganese and chromium metals can be derived from low grade ores. Electrolytic manganese is now produced at a rate of 3600 tons per year, and a plant to produce 6000 tons of electrolytic chromium yearly is under construction. As a result of lower cost and much higher purity, these electrolytic metals are likely to displace those now made by aluminothermic reduction of the oxides. The pure metals are used chiefly in nonferrous alloys, especially those having high strength and corrosion resistance at high temperatures. The cost of electrolytic chromium has not been established by commercial production. If it can be sold for 30¢, the present price of electrolytic manganese, it may become competitive with low-carbon ferrochrome.

The Boulder City laboratory also has shown that battery-grade manganese dioxide and chromate chemicals can be produced from domestic ores at efficiencies only slightly less than can be attained with imported ores used for those purposes. A pilot plant designed to concentrate 50 tons of Artillery Peak manganese ore per day has been constructed, and flotation tests are now in progress. Concentrates will be upgraded with manganese oxide recovered from flotation middlings by a dithionate leaching process. A caustic leach to remove silica will be tested also. Upgraded products will be nodulized to provide consolidated material suitable for charging into an electric smelting furnace.

Such combinations of ore dressing and chemical treatment have been regarded as the only feasible means of utilizing low grade ores that do not yield standard grade products by ore dressing alone. However, chemical beneficiation is so costly in relation to the price of imported ores that it should not be regarded as an economic upgrading step in the production of ferroalloys. Also, installation of chemical treatment plants with enough capacity to provide all the ferroalloys required by the steel industry would take more time and effort than could be spared in an emergency.

Ferroalloy producers naturally demand the best available ores because the higher cost of smelting low grade ore usually will more than offset the lower cost of inferior ore. However, if ferroalloy producers were forced to use low grade ores, they would discover that, in most instances, direct smelting after ore dressing would cost less than chemical treatment followed by smelting.

Current specifications for metallurgical ore are based upon the best grades of ore and should not be considered definitive. They should be disregarded in any effort to find the best method for producing ferroalloy from a low grade ore. One specification regarded as critical is the ratio of manganese or chromium to the iron content of an ore. This ratio to a great extent determines the manganese or chrome content of the ferroalloy and also has some effect on smelting cost and furnace capacity in terms of the manganese or chrome content of the alloy.

The steel industry has become accustomed to certain grades of ferroalloys, and it has been assumed generally that alloys having much higher iron content would not be acceptable. One reason advanced for this view is the chilling effect of extra iron when the ferroalloy is added to the ladle.

Metallurgical chrome ore, normally with 48 pct

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Cr_2O_3 and a chrome-iron ratio of 3, yields a ferrochrome containing about 70 pct chromium. Concentrates obtained from Oregon black sands and from low grade Montana chromite contain only 40 pct Cr_2O_3, and the chrome-iron ratio is about 1.5. In the Bureau of Mines laboratory at Boulder City, 5 tons of high-carbon ferrochrome, containing about 55 pct chromium, were made from these off grade concentrates and shipped to the Republic Steel Corp. for tests in open hearth furnace.

Six 100-ton heats of alloy steel were made, using the special ferrochrome obtained from the Bureau of Mines. The average recovery of chromium was 84.9 pct, comparing favorably with that obtained from commercial alloys. Microscopic examination of nonmetallic inclusions and etch tests on polished sections of rolled billets revealed acceptable material from all of the heats.

The GSA price of $115 per ton for high grade lump chromite is subject to a bonus or penalty for variations from the standard grade. Montana chrome concentrates do not meet minimum specifications; but, if the scale of penalties were extended, the GSA price would be $41 per long ton for 40 pct Cr_2O_3, with a 1.5 ratio, or 6.8¢ per lb of contained chromium.

Assume that, by a chemical process, chromic oxide is recovered from a portion of this ore and composited with the amount of ore concentrate required to obtain a chrome-iron ratio of 3. Briquets made from this mixture would contain 57 pct Cr_2O_3; they would be a superior source of standard grade ferrochrome; and, at GSA rates, would bring a price of $151 per long ton, equivalent to 17.3¢ per lb of chromium.

The highest quality imported chromite ore (48 pct Cr_2O_3) sells for $54 per long ton, or 7.5¢ per lb of chromium. High-carbon ferrochrome is quoted at 21¾¢ per lb of chromium. Compare these prices with GSA schedules that offer 17.3¢ per lb of chromium in upgraded briquets, 16.0¢ per lb chrome in standard grade ore, and an extrapolated value of 6.8¢ per lb chromium in a concentrate that does not meet minimum specifications.

During the last war, chromite concentrates were made from Montana ores at reported costs of $35 to $39 per ton, including mining and milling royalties, amortization and trucking to Columbus, Mont. These costs cover only short operating periods at less than optimum capacity and would have been considerably lower in a continuing operation at full capacity. Even with present labor costs, it should be possible to produce concentrates for $35 per long ton, or 6¢ per lb of chromium.

Cost estimates indicate that these chromite concentrates can be upgraded chemically for about 6¢ per lb of chromium and then smelted to ferrochrome for an additional 8¢ per lb of chromium. If the concentrates can be produced for 6¢ per lb of chromium, the cost of ferrochrome will be about 20¢ per lb of chromium.

These same estimates indicate that costs for nodulizing and smelting mill concentrates will not exceed the cost of smelting high grade ore by more than 2¢ per lb of chromium in the alloy. The difference may be less, and it is much smaller than the cost of chemical upgrading or the 10.5¢ bonus offered by GSA. With the same cost for the crude concentrate, the ferrochrome will cost about 16¢ per lb of chromium. This is well below the market price for commercial alloy and even less than the GSA price (17.3¢) for briquets made from upgraded concentrates.

There is no doubt that the steel industry can use 55 pct ferrochrome made by smelting crude concentrates and for most uses would prefer this alloy, if the standard grade could be obtained only at a higher cost. In an emergency, enough 55 pct ferrochrome to supply a large part of requirements can be produced from Montana chromite concentrates in existing furnaces on short notice and at reasonable costs. If mining, milling, and smelting are efficiently integrated in units having large capacities, competitive production of ferrochrome from these ores may be possible.

The same general considerations apply to upgrading inferior manganese ores, and the pilot plant at Boulder City will provide data that can be used to estimate costs on various treatments of Artillery Peak ore. The cost of nodulizing and smelting a flotation concentrate containing 30 to 35 pct manganese will be compared to the cost of chemical beneficiation and smelting of the upgraded product.

Artillery Peak ore has a satisfactory manganese-to-iron ratio, and a standard grade of ferromanganese can be made by direct smelting. Except for lead and phosphorus that must be eliminated before smelting, silica is the chief impurity. Lime must be used to flux the silica, and the loss of manganese may be high on account of the increased amount of slag. However, the high silica will be an asset if manganese ferrosilicon is produced.

At the Bureau of Mines Pittsburgh station, a small blast furnace is used to recover a high-phosphorous spiegeleisen from open hearth waste slag. This spiegel is selectively oxidized in a basic-lined bessemer converter to make a slag containing 55 to 60 pct manganese with a residual metal containing up to 5 pct phosphorus. The converter slag is smelted to 80 pct or higher ferromanganese. Utilization of the high-phosphorus residual molten iron is a problem that must be solved before the overall operation can have commercial applications.

The large amount (7 million tons) of open hearth slag being discarded annually as waste has aroused considerable interest in the possibility of recovering ferromanganese from a material containing 7 to 10 pct manganese. In comparison with this lean material, ores containing 30 to 35 pct manganese must be regarded as relatively high grade, and it would appear that an increased degree of attention should be given to the possibility of direct smelting this potential source of manganese.

There is no magic formula by which a low grade ore can be converted to a marketable product at a cost that does not exceed the cost of producing the same product from a high grade ore. The difference may be relatively small if a product having high unit value is obtained by chemical or electrolytic methods. The grade of ore has more influence on costs if a ferroalloy is produced by smelting. If the low grade ore must be chemically upgraded before smelting, treatment costs will be much higher than the cost of smelting high grade ores.

The grade of ore and mining, milling, and transportation costs must all be compared with the grade and costs of imported ore in estimating economic possibilities of treating ore from any specified deposit. For competitive production of ferroalloys, the milled ore must yield an acceptable alloy by direct smelting, and it must cost less per unit of metal than high grade imported ores when adequate supplies of the latter are available.

Evaluation of the pH and Conductivity Methods of Slag Control

by P. D. S. St. Pierre

IN recent years the use of high sulphur fuels and charges in steelmaking has necessitated rapid methods of slag control in order to insure the production of high quality steel. Several systems of control have been discussed in the literature, and of these the method described by Philbrook, Jolly, and Henry[1] has received the most attention recently. In their process the hydrogen ion concentration of an aqueous slag suspension is determined under controlled, but arbitrary conditions. The basicity of the slag may then be derived from a previously established pH-*V* ratio curve. Thus, by measuring the pH, some indication of the *V* ratio is gained, from which significant properties of the slag may be deduced. Philbrook et al. made no claims as to the theoretical justification of their treatment, but simply offered it as a rapid means of slag control. Encouraging results caused others to study the method and publish their findings.[2-5] Although there was fair agreement on the efficacy of the method, no common parameter was evolved against which pH readings could be plotted. The results of the various determinations were related to such *V* ratios as CaO/SiO_2 and $CaO/SiO_2 + P_2O_5$ by some,[1-3] while others[5] plotted against "excess lime." Rivet,[4] however, found a simple proportionality between the hydrogen ion concentration and the sulphur content of the molten steel beneath the slag. Since slag control in North American practice is largely directed toward the efficient removal of sulphur from steel, Rivet's approach to the problem had the advantage of connecting directly a property of the slag with the impurity being removed.

In view of the diversity of parameters used it seemed probable that some fundamental point was being overlooked in seeking a relationship. Examination of the literature revealed that much emphasis had been laid on experimental detail but scant attention paid to the fundamental aspects of the hydrolysis of the aqueous slag suspension. Consequently the present investigation was undertaken with the object of rectifying this deficiency and deriving, if possible, a parameter of general application.

Petrographic studies on several matured Canadian open-hearth steel slags have shown that their major constituents are dicalcium and tricalcium silicates set in a complex matrix of iron oxides and ferrites. These phases are shown in Figs. 1 and 2. Free lime is frequently encountered in slags, but is not an equilibrium phase since on continued heating it dissolves forming silicates or ferrites. Thus, it appeared that a study of the hydrolysis of the major phases in steel slags would be profitable.

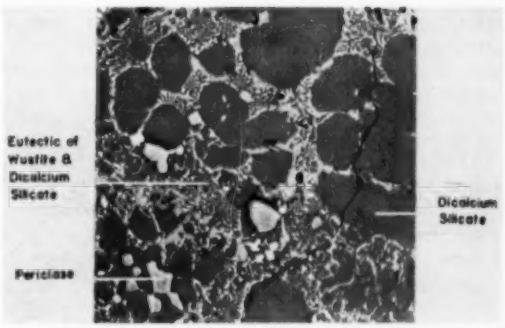

Fig. 1—Polished section of a typical basic open hearth slag. X400. Area reduced approximately 75 pct for reproduction.

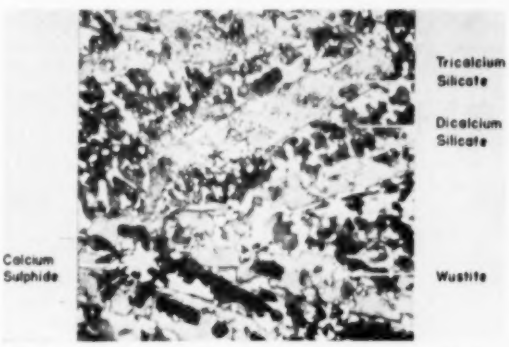

Fig. 2—Polished section of a typical basic open hearth slag. X500. Area reduced approximately 75 pct for reproduction.

The experimental technique used in the present work was a modification of that described by the other workers.[1-5] Electrical conductivity measurements were also made on the suspensions, since Smith et al.[5] had claimed advantages for this method in their paper on slag control.

Experimental Procedure

The compounds studied in this investigation were prepared in the laboratory by heating purified oxides

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Table I. The pH and Conductivity Values of Aqueous Suspensions of Steel Slag Phases

Compound	Formula	pH	Conductivity, Microhm at 20°C
Wollastonite*	$CaO \cdot SiO_2$	10.7	123
Rankinite*	$3CaO \cdot 2SiO_2$	11.0	192
Dicalcium silicate	$2CaO \cdot SiO_2$	11.4	681
Tricalcium silicate	$3CaO \cdot SiO_2$	11.9	1810
Lime	CaO	12.6	7800
Fluorite	CaF_2	6.7	60
Monocalcium ferrite	$CaO \cdot Fe_2O_3$	9.0	46
Dicalcium ferrite	$2CaO \cdot Fe_2O_3$	10.5	124
Periclase	MgO	10.1	98

* Not normally found in steel slags, but included in the series to show development of hydrolysis with increasing lime content.

(or carbonates) of the various components to suitable temperatures in platinum crucibles. Samples freshly ground to −100 mesh were used throughout the investigation, the weight taken being varied according to the purpose of the run. After weighing to ±0.01 g accuracy, the samples were mechanically shaken for a specified time at constant temperature in 100 cc of CO_2-free distilled water. Prior to pH and conductivity determinations, slag suspensions were filtered on Whatman 42 filter papers which previously had been thoroughly washed with CO_2-free distilled water. The first 25 cc of the filtrates was used to wash the electrodes of the instruments, the remainder was used for the actual determinations.

Results

Table I shows the pH and conductivity of the leached elements resulting from shaking 0.50 g of possible open-hearth steel slag phases individually in 100 cc water for 30 min. It should be emphasized that the results relate only to determinations made under the conditions stated and therefore they do not necessarily represent equilibrium values. The pH of wüstite (FeO) was not determined, because it is very difficult to prepare pure ferrous oxide. Although several attempts were made, no really satisfactory product was obtained. There is, however, good reason to believe that its pH would be relatively low, since that of ferrous hydroxide is quoted as only 9.5 in saturated solution. If wüstite caused any pH change at all under the conditions of experiment, it would probably first form hydroxide, in which case it is reasonable to suppose the final pH would not exceed 9.5.

Table II. The pH as a Function of Weight of Steel Slag Phases in Aqueous Suspension

Compound	Formula	Weight, G	pH
Dicalcium silicate	$2CaO \cdot SiO_2$	0.05	10.8
		0.10	10.9
		0.25	11.1
		0.50	11.4
		1.00	11.5
		2.00	11.5
Tricalcium silicate	$3CaO \cdot SiO_2$	0.05	11.5
		0.10	11.7
		0.25	11.8
		0.50	11.9
		1.00	12.0
		2.00	12.1
Lime	CaO	0.01	11.5
		0.025	11.9
		0.10	12.4
		0.20	12.6
		0.30	12.5
		0.50	12.6

It will be seen from Table I that the lime and calcium silicates are the most easily hydrolyzed compounds and their presence may therefore be considered as the controlling factor in determining the pH of an aqueous slag suspension. Thus, in a mixture of dicalcium and tricalcium silicates the pH will be controlled by the tricalcium silicate, provided there is sufficient quantity present to exceed the maximum pH given by dicalcium silicate. If the tricalcium silicate is present in quantities which exceed that required to produce its maximum pH, there is no means of determining the excess since the pH remains constant.

Because, from a hydrolysis point of view, lime, dicalcium and tricalcium silicates are the most important compounds in a steel slag, data on their hydrolysis as a function of weight were determined. Such data are given in Table II, and the information is shown graphically in Fig. 3. The standard experimental procedure described previously was used to obtain the data. A shaking time of 30 min was found to yield steady pH readings for all the compounds.

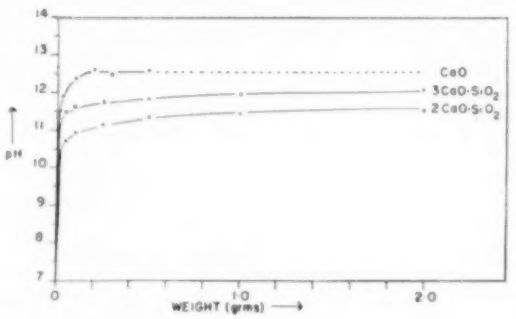

Fig. 3—Effect of weight on the pH of various slag phase aqueous suspensions.

For a given weight of sample the pH increases rapidly in the first few minutes, but after a shaking time of 10 min the rate of increase of pH is very low. Thus, by using 30 min periods of shaking the time factor may be ignored completely.

The curves shown in Fig. 3 immediately reveal the limitations of the pH method of slag control. It will be observed that the curves fall into two parts: at first, pH rises very rapidly with increasing weight of sample, but later the rate of increase is very small. Since slag control implies determination of the calcic mineral content of the slag, pH determinations in general will be either under or over sensitive to variations in such minerals. The choice of weight of slag sample is therefore an important factor in determining the sensitivity of the method. Some workers have used a 0.25 g sample in their investigations, while others have used 0.50 g of slag. Thus in a typical slag containing 30 to 50 pct dicalcium silicate, those using the 0.25 g sample would find the method sensitive to the weight factor and those using the 0.50 g sample would find it insensitive. Clearly in selecting the weight of sample, consideration must be given to the amount of calcic mineral likely to be encountered. If free lime is present it will be seen from the graph that little can be done, since traces of this oxide have considerable effect on the pH of the suspension. If the slag is in equilibrium free lime is generally absent, but in practice it is often found as undissolved material.

The pH of a suspension is controlled by the constituent which will cause the highest pH, and this is a function not only of the intrinsic chemical properties of the compound but also of the quantity present. This latter point may be illustrated by the data given in Fig. 3. It will be seen, for example, that 0.05 g of tricalcium silicate yields almost the same pH as 0.5 g of dicalcium silicate, and there is no way of telling from the pH determination alone which compound is predominant in its effect. If it is known at what stage of the heat the sample was taken, a fairly good guess may be made, for only the late slags are likely to contain tricalcium silicate, see Figs. 1 and 2.

Once the maximum pH of the predominating compound has been reached no determination of the quantity present can be made, for further increases have no effect on the pH. In practice this condition exists as soon as the flat part of the pH-weight curve is reached.

In the case of practical slags a complication exists in that the pH attained by the aqueous suspension will be a function of the surface of mineral exposed, since the calcic minerals may be coated by wüstite (FeO), etc., and can react only through exposed surfaces. This shielding factor may prevent the mineral from hydrolyzing to the value expected for the weight present.

This discussion provides some idea of the inherent difficulties of the pH method of slag control. It has been shown that the mode of hydrolysis of the calcic minerals prevents the hydrogen ion measurement from being an accurate method of estimating the amounts present.

Having shown that the alkalinity of slag suspensions depends on the hydrolysis of certain minerals, it will be apparent that there is no theoretical foundation for relating pH to parameters such as "V ratio" or "excess base." The partial success obtained with some of these parameters might be explained by the fact that they are, to some extent, a crude measure of the calcic minerals present in the slag. Such parameters can therefore be related to pH variations only in a general way.

The information given in Fig. 4 and Tables I and III shows that the conductivity method of control suffers from much the same disadvantages as the pH method.

The conductivity curves of the various compounds are more widely separated than the pH curves and this makes it easier to distinguish between the several phases present in a slag. Equally, however, it will be seen that very minute quantities of lime present in the slag can upset measurements very considerably. Unfortunately the slopes of these curves render them unsuitable for measuring the amounts of phases present in suspensions. In this respect the conductivity method of control has no advantage over the pH technique.

Table III. Conductivity as a Function of Weight of Steel Slag Phases in Aqueous Suspension

Compound	Formula	Weight, G	Conductivity, Microhm at 20°C
Dicalcium silicate	$2CaO \cdot SiO_2$	0.10	300
		0.25	475
		0.50	681
		1.00	818
		2.00	914
Tricalcium silicate	$3CaO \cdot SiO_2$	0.10	1200
		0.25	1420
		0.50	1810
		1.00	2530
		2.00	3560
Lime	CaO	0.01	707
		0.025	1606
		0.10	5650
		0.20	7800
		0.30	7840
		0.50	7800

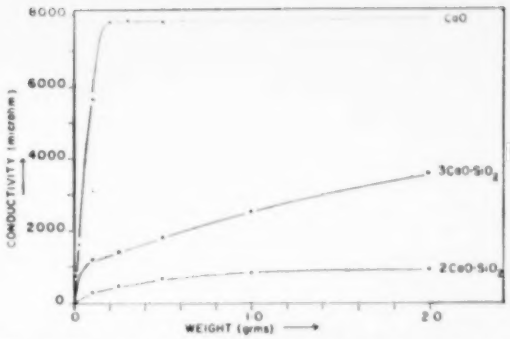

Fig. 4—Effect of weight on the electrical conductivity of various slag phase aqueous suspensions.

Discussion

Although pH and conductivity measurements can indicate in a general way the mineralogical development of a slag as the heat proceeds, the nature of hydrolysis of the calcic minerals precludes exact quantitative measurements. In plant practice the method might, however, be used to check the type of slag being made (i.e., whether dicalcium or tricalcium silicate is present or not), provided no free lime is present, and in this application the conductivity measurement would be superior. Thus the pH or conductivity of a good steel slag could be checked and thereafter the composition of all slags adjusted to yield similar results.

Conclusions

1—It is improbable that the pH or conductivity methods of slag control can be improved by refinement of technique or more exact interpretation of results, since both methods are subject to variable sensitivity.

2—The presence of traces of undissolved lime in slag samples has a considerable effect on pH or conductivity measurements.

3—There is little theoretical justification for relating pH or conductivity results to the "*V* ratios" or "excess base" used by previous workers.

Acknowledgment

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Density and Hydrogen Occlusion of Some Ferrous Metals

by J. H. Keeler and H. M. Davis

Densities of SAE 1020 (I) and ingot iron (II) decreased with cold rolling to minima at 60 pct reduction, whereas high-purity iron (III) was unaffected. I recovered promptly with annealing; II, sluggishly and incompletely. Hydrogen occlusion of III or II was barely affected by 60 pct cold reduction, but that of I was increased a hundredfold.

IT has been reported of iron that its density is decreased and its occlusive capacity for hydrogen is increased by cold work. One of the most interesting aspects of each of these properties is that the changes brought about by cold work are not necessarily eliminated by annealing and recrystallization. Yet recrystallization is generally accepted as restoring to their original condition the properties or characteristics altered by cold work.

A number of investigators have reported a decrease in density with cold reduction. Two[1, 2] have shown that annealing after cold reduction may not return the density to its original value. It has been suggested[1] that cracks or flaws in inclusions produced during cold drawing contribute to the lowering of the bulk density of the metal. Extremely high-purity copper has been reported[2] as not exhibiting a density decrease with cold reduction. On the other hand, calculations[1] indicated that grain distortion by itself can produce expansion of the order of magnitude of the changes observable. Maier[3] showed that both the density of iron and that of copper passed through a minimum with increasing cold reduction.

Although much information is available on the subject of hydrogen in steel[4-7] and it is well known that plastic deformation of a metal may increase its occlusive capacity,[8-12] most of the reports pertain to charging the metal with hydrogen by cathodic deposition or with acid. Darken and Smith,[12] who employed acid charging, emphasized the great influence of cold work on the occlusive capacity of iron for hydrogen and showed the inability of a prolonged high-temperature annealing treatment to restore the occlusive capacity of cold-worked steel to the value exhibited in the hot-rolled condition.

It has been reported[13] that an "endothermic" metal* in the cold-worked condition can be surcharged when exposed to gaseous hydrogen. Thorough annealing,

Table I. Chemical Compositions of Materials Investigated

Element	Percentage of Element Present		
	Ingot Iron	SAE 1020	High-Purity Iron
Carbon	0.015	0.17	0.006
Manganese	0.06	0.33	<0.01
Phosphorus	0.004	0.009	0.002
Sulphur	0.025	0.022	0.002
Silicon	0.001	0.024	0.030
Aluminum	0.003	0.004	<0.005
Oxygen	0.066	0.002	0.008
Nitrogen	0.004	0.001	<0.001
Copper	0.04	0.016	<0.005
Nickel	0.018	0.016	0.014
Chromium	0.008	0.010	<0.005
Molybdenum	0.004	0.002	<0.005
Cobalt	0.007		
Tin	0.009	0.001	<0.005
Arsenic	0.006		Faint trace
Titanium	0.0		<0.008
Zirconium			Very faint trace
Magnesium			Trace
Lead	Trace		Trace
Strontium			Very faint trace

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* Endothermic metal refers to metals like iron, nickel, copper, and platinum, which occlude hydrogen with absorption of heat, and for which the occlusion increases with rising temperature.

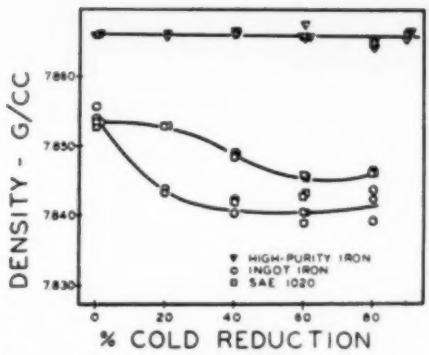

Fig. 1—Relation between density and percentage of cold reduction. The high-purity iron showed no noteworthy decrease in density.

on the other hand, has been said[14] to give to iron at slightly elevated temperatures no "detectable capacity" for absorbing hydrogen. Apparently the influence of cold reduction has been found up to the recrystallization temperature.[16] Tammann[15] has observed considerable evolution of hydrogen from surcharged iron at the recrystallization temperature during heating.

The explanation for the influence of strain (plastic) in the metal upon occlusive capacity is controversial. Whether the hydrogen in molecular form is stored in rifts or lattice imperfections as suggested by Smith[17] and coworkers, and by Zapffe, Sims, and coworkers,[18] or whether hydrogen atoms or protons are held by the lattice imperfections in some manner, has not been fully resolved.

Only indirect evidence has been found showing the influence of cold work or strain in iron on the occlusive capacity for hydrogen at elevated temperatures.

Table II. Possible Relation of Area to Density Loss in Annealing

	Area/Volume	Density Loss x 10^4/Volume
Hot-rolled high-purity iron annealed at 1200°C	19.6	21
Cold-reduced high-purity iron annealed at 1200°C	48.0	40

Experiment

Ferrous metals of three compositions were studied in this investigation: a low-metalloid rimmed ingot iron; a rimmed steel, nominally of SAE-1020 grade; and a high-purity iron. The first two were sheets of hot-rolled commercial tonnage alloys, whereas the third was obtained as a 10 lb ingot from the National Research Corp. The chemical compositions are given in Table I.

The heat treatments were done in purified helium in an airtight furnace. The densities were obtained by use of Archimedes' principle, the liquid being monobromobenzene of known density. The overall error was calculated to be possibly as large as 0.00056 g per cc, although separate density calculations for a given specimen have agreed within 0.0002 g per cc. However, the value of 0.00056 g per cc is somewhat less than the variation occasionally found between individual samples of the same material given identical treatments.

The hydrogen occlusion was determined by a technique similar to that of Sieverts. An experiment consisted of heating a metal specimen in an atmosphere of hydrogen at a constant pressure of 710 mm of mercury and measuring over mercury the equilibrium volume obtained at various temperatures. These values were then compared with the volumes obtained at the same temperatures during a blank experiment in which no hydrogen was absorbed. The difference between the volume obtained with metal present and the corresponding value found with the blank showed the amount of hydrogen absorbed by the metal at the particular test temperature.

Results and Discussion

The experiments revealed that neither the density nor the hydrogen occlusion of a ferrous material is necessarily altered appreciably by cold reduction or by subsequent annealing. Each material must be considered individually.

High-Purity Iron: The density of the hot-rolled high-purity iron was found to agree very well with the values cited by Cleaves and Thompson,[19] who reported an average of 7.867 g per cc as obtained by direct determination. The value found in this investigation was 7.866 g per cc. (A density of 7.874 g per cc for exceptionally high-purity iron has been reported by Cleaves and Hiegel.[20])

Contrary to the widely accepted opinion that cold working decreases the density of a metal, it was found that sheets of high-purity iron cold-rolled 20, 40, 60, 80, and 90 pct from the hot-rolled condition did not undergo any noteworthy decrease in density, Fig. 1. In no case did the density of this material increase with annealing after cold reduction, Fig. 2. Actually, annealing in helium at a temperature of 1200°C decreased the density, a change which had not been foreseen.

Specimens of hot-rolled high-purity iron (about 0.10 in. thick), annealed with the above material at 1200°C, showed a small decrease in density, although not so large a loss as the thinner, cold-reduced specimens. If the density decrease at 1200°C resulted from a surface phenomenon such as volatilization or reaction with an impurity in the helium, a smaller density loss would be expected for the thicker material than for the thinner sheets. A consideration of the area-to-volume ratios and the density-loss-to-volume ratios of the hot-rolled and the cold-reduced high-purity iron specimens suggested that a surface phenomenon was involved, Table II.

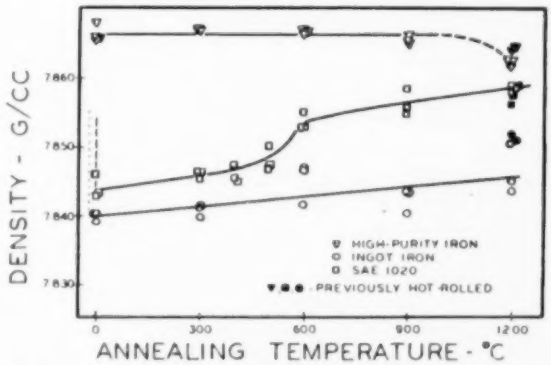

Fig. 2—Relation between density and annealing temperature. The SAE 1020 exhibited a rapid increase in density during annealing at 600°C. The high-purity iron and the ingot iron showed a density decrease after annealing at 1200°C.

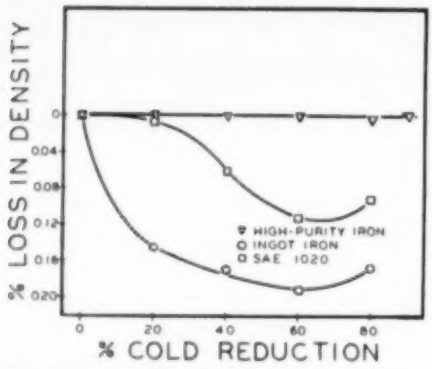

Fig. 3—Relation between percentage loss in density and percentage cold reduction.

Table III contains data showing the lack of influence of either cold reduction or annealing upon the density of the high-purity iron with the exception of the anneal at 1200°C.

Ingot Iron: The density of the ingot iron was found to decrease with increased cold deformation up to 60 pct reduction, as shown in Figs. 1 and 3. A slight increase in density was observed at 80 pct reduction, which is in agreement with Maier.[5]

The density determined for the hot-rolled ingot iron was nearly the same as that obtained for the hot-rolled SAE 1020, Table II. On the basis of purity, it might be argued that, because of the higher oxygen content, the volume of inclusions in the ingot iron would be the greater, giving that material the lower density. It was also suspected that the commercially hot-rolled ingot iron contained some strain, so that the metal was not of maximum density.

The recovery of density by the ingot iron with annealing after cold reduction showed a greater spread in values than was observed in the other materials. Careful rechecking indicated that the measured densities were correct for the individual specimens. Apparently the ingot iron was of a lower degree of homogeneity than were the other metals. In spite of the spread in the data, the recovery of density was clearly shown to be relatively small, inasmuch as the greatest recovery was only about 42 pct of the decrease resulting from cold reduction.

As did the high-purity iron, the hot-rolled ingot iron suffered a density decrease when annealed at 1200°C. The magnitude of this loss was 0.0039 g per cc.

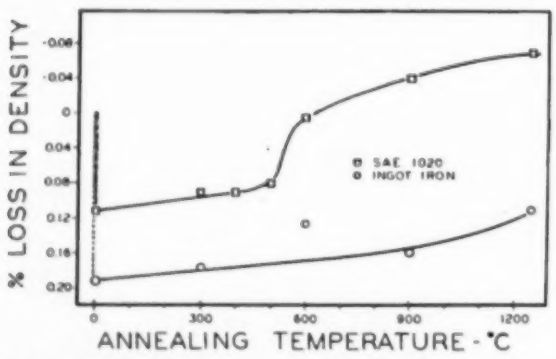

Fig. 4—Relation between percentage loss in density and annealing temperature.

The density of the cold-reduced ingot iron after the 1200° anneal was only slightly increased over that found after the 900° anneal. This may have been the resultant of a density loss (also shown by the hot-rolled material) superimposed on a further recovery from the effects of cold reduction. If it is assumed that the same ratio obtains between the density losses of the hot-rolled and the cold-reduced ingot iron after the 1200° anneal as was observed for the hot-rolled and the cold-reduced high-purity iron, then a value close to the observed density of the unannealed, hot-rolled ingot iron can be calculated, Table IV.

When the ratios in Table IV are assumed equivalent,

$$\frac{0.0021}{0.0040} = \frac{0.0039}{x}, \text{ and } x = 0.0074 \text{ g per cc.}$$

If the 0.0074 g per cc is added to the experimental density of the ingot iron as cold-reduced and annealed at 1200°C,

$$0.0074 + 7.8463 = 7.8537 \text{ g per cc}$$

the sum is close to the observed density of the hot-rolled ingot iron.

SAE 1020: Although the density of the SAE 1020 decreased as the cold reduction was increased, the rate of density decrease was not so rapid as was that for the ingot iron. The minimum density was reached after a cold reduction of 60 pct and, as with the ingot iron, a slight increase in density above the minimum was observed after a cold reduction of 80 pct, Figs. 1 and 3.

Annealing of the 60 pct cold-reduced SAE 1020 at 300°C for 2 hr provided negligible recovery of density. At a temperature of 400°C an unmistakable increase in the density was found and, after the anneal at 600°C, density recovery was substantially complete. Higher annealing temperatures, 900° and 1200°C, increased the density to a value even greater than that of the nominally hot-rolled condition, Figs. 2 and 4. It was evident from the microstructures of these specimens that little carbon, if any, remained in the metal after the anneal at either 900° or 1200°C. If a straight-line relationship is assumed between the density of cementite, 7.67 g per cc,[21] and the density of high-purity iron, 7.87 g per cc, the influence of carbon upon density in the SAE 1020 containing 0.17 pct C is approximated at 0.005 g per cc. If 0.0050 g per cc is subtracted from the density of the hot-rolled SAE 1020 annealed 2 hr at 1200°C (7.8574 g per cc), a value of 7.8524 g per cc is obtained. The original hot-rolled and not decarburized material had a density of 7.8534 g per cc, which agrees well with the value obtained by correction for carbon.

The anneal at 1200°C apparently did not produce in the SAE 1020 the density decrease found in the commercially pure ingot iron and in the high-purity iron. The hot-rolled SAE 1020 agreed closely in density with the 60 pct cold-reduced SAE 1020 after both had been annealed at 1200°C.

It was reported previously that the high-purity iron exhibited no decrease in density with cold reduction of as much as 90 pct by rolling. This behavior is contrary to the theoretical prediction that crystal deformation alone can produce a density decrease of the experimentally familiar magnitude. With the same kind of deformation, however, the ingot iron and the SAE 1020 did exhibit a loss of density.

If a decrease in density with cold reduction can be associated with the presence of foreign elements in the metal, a plausible explanation of that relationship is that imperfect or uneven flow of metal occurs at phase boundaries during plastic deformation. Voids in the metal adjacent to inclusions could possibly serve in explanation of the observations.

The presence of voids in cold-worked iron or steel has been indicated by the blistering of such materials by hydrogen after acid pickling. It is also widely observed that such blisters often occur in the vicinity of inclusions in the metal. It is possible that the inability of the metal, during plastic deformation, to flow perfectly about small inclusions would both cause the voids necessary to initiate blistering and contribute to a decrease in the density of the metal.

The wide difference in density recovery with annealing of the SAE 1020 and the ingot iron suggests that different mechanisms are involved in the density decrease induced in these two metals by cold work. It would be instructive to determine the influence of various phases upon the plastic behavior of the surrounding ferrite lattice.

Hydrogen Occlusion: The first experimental values obtained for the occlusion of hydrogen by high-purity iron and by ingot iron in the hot-rolled, the 60 pct cold-reduced, and the annealed condition did not differ significantly from each other. Because this lack of influence of cold work was not anticipated by information found in the literature, several occlusion tests with annealed nickel sheet were conducted to show whether the system was sufficiently sensitive to pick up notable differences in hydrogen occlusion. Inasmuch as the values obtained for the hydrogen occlusion by nickel agreed very well with the data of Armbruster,[22] confidence in the measurement was established.

The continued study of the occlusion of hydrogen revealed no large variations in the occlusive capacity of the high-purity iron or of the ingot iron, whether the metal was in the hot-rolled, the 60 pct cold-reduced, or the annealed condition, Figs. 5 and 6. The occlusive capacity of the SAE 1020 in the hot-rolled and in the annealed condition was similar to that of the high-purity iron and that of the ingot iron, Fig. 7. But the 60 pct cold-reduced SAE 1020 exhibited a phenomenal occlusive capacity for hydrogen at the lower test temperatures. At 250°C this material occluded 9.03 cc of hydrogen per 100 g of metal, about 24 hr being necessary for the attainment of equilibrium. This volume was approximately one hundred times the amount taken up by the hot-rolled SAE 1020, or by either of the other two irons. At 300°C, the occlusion of hydrogen by the cold-reduced SAE 1020 fell to 3.84 cc per 100 g. Here, less than 30 min was necessary for the establishment of equilibrium. A minimum occlusion was reached at 450°C, where 1.50 cc per 100 g was taken up, and the normal solubility rise with a further increase of temperature was observed at 550°C. As the metal was cooled, the curve of occlusion vs temperature did not follow the same path that it took during heating. Apparently some recrystallization (among other possible changes) had occurred at the higher test temperatures, diminishing the occlusive capacity. During cooling, a broad minimum in the hydrogen occlusion occurred at 350° to 400°C, and the amount of occlusion increased slightly at 300° and 250°C. The hydrogen occlusion of the cold-reduced SAE 1020 plotted against temperature is shown in Fig. 8, which also includes, for comparison, selected values from the literature and from this investigation for the occlusive capacity of nickel and of iron.

The density decrease and the large increase in hydrogen occlusion which were exhibited by the

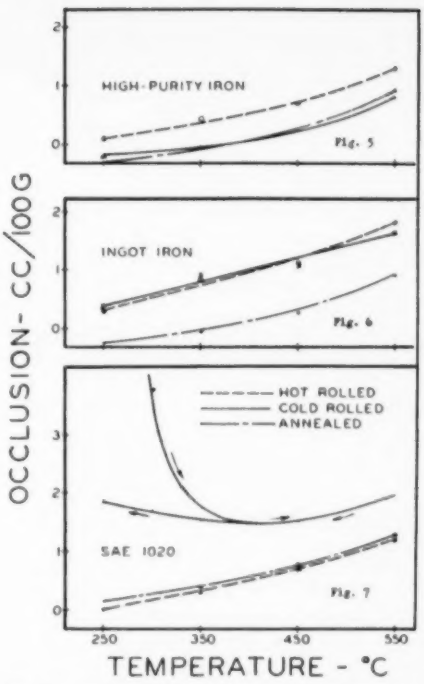

Fig. 5—Hydrogen occlusion vs temperature for high-purity iron in various conditions.
Fig. 6—Hydrogen occlusion vs temperature for ingot iron in various conditions.
Fig. 7—Hydrogen occlusion vs temperature for SAE 1020 in various conditions. The curve for the cold-reduced material is incompletely shown here. See Fig. 8.

Table III. Densities of Test Materials in Various Conditions

Condition	Density, G per CC at 25°C		
	High-Purity Iron	Ingot Iron	SAE 1020
Hot-rolled	7.8662	7.8549	7.8534
Cold-reduced:			
20 pct	7.8664	7.8435	7.8530
40 pct	7.8663	7.8415	7.8488
60 pct	7.8664	7.8399	7.8445
80 pct	7.8650	7.8418	7.8465
90 pct	7.8664		
60 pct cold-reduced plus:			
2 hr at 300°C	7.8671	7.8409	7.8460
2 hr at 400°C			7.8459
2 hr at 500°C			7.8470
2 hr at 600°C	7.8666	7.8450	7.8537
2 hr at 900°C	7.8655	7.8424	7.8564
2 hr at 1200°C	7.8622	7.8463	7.8587
Hot-rolled plus:			
2 hr at 1200°C	7.8641	7.8511	7.8574

In most instances these values are averages from three specimens in each condition, three determinations per specimen.

Table IV. Comparison of Density Losses in Annealing

Metal	Condition Before 1200°C Anneal	Annealing Loss, G per CC
High-purity iron	Hot-rolled	0.0021
	Cold-reduced	0.0040
Ingot iron	Hot-rolled	0.0039
	Cold-reduced	x

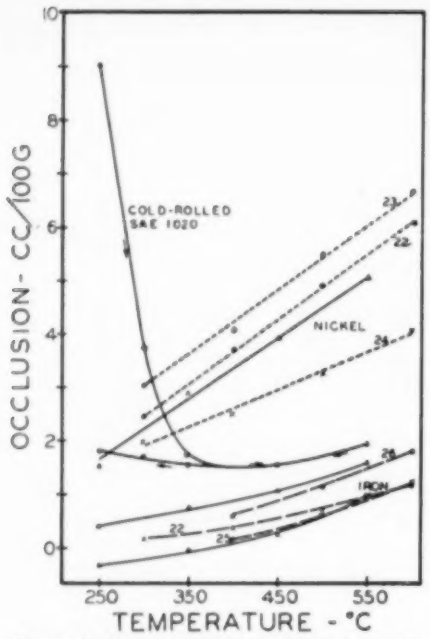

Fig. 8—Hydrogen occlusion vs temperature for nickel (dotted lines 22, 23, and 24) and for iron (dashed lines 22, 25, and 26). The numbers refer to references which are the original sources of the data indicated by broken lines. The solid lines are data from this investigation. The data for iron from this research indicate the wide spread of values. The curve for the cold-reduced SAE 1020 is in remarkable contrast to the other curves.

SAE 1020 after cold reduction were not compatible with the concept of the storage of molecular hydrogen. Approximately 2.9 cc of gas was charged into 4.1 cc of this steel at 250°C, the source of the hydrogen being the gas at a pressure slightly less than 1 atm. Since the observed density decrease of 0.18 pct in this material could provide a calculated storage volume of only 0.00524 cc, storage as hydrogen gas would require a pressure of 516 atm in the voids, or 552 times the charging pressure. Yet the dissociation which makes charging possible at the test temperature, 250°C, would hardly permit the development of void pressures any greater than 1 atm.

The hydrogen-occlusion results for the three ferrous materials in various conditions and for annealed nickel were obtained by subtraction of the observed volumes from the average of the appropriate blank values. Because the blank could vary by as much as ±1.75 pct, the very small occlusive capacity of iron for hydrogen, particularly at 250°C, occasionally produced a fallacious negative value for occlusion. Obviously, in these instances the slight occlusion of hydrogen was less than the experimental error.

Summary and Conclusions

The high-purity iron showed neither a density decrease nor any noteworthy change in hydrogen occlusion with cold reduction or annealing. Apparently the presence of impurities is necessary for a density decrease with cold reduction.

Cold reduction decreased the density of ingot iron but did not change the hydrogen occlusion of the metal. Annealing after cold reduction restored only 42 pct of the original density loss and did not influence the hydrogen occlusion.

Cold reduction decreased the density of SAE 1020 and increased the hydrogen occlusion at 250°C about 100-fold. This remarkable hydrogen occlusion decreased at higher temperatures, but remained greater than that of the unstrained material. Annealing after cold reduction eliminated the density loss and diminished the large occlusive capacity caused by cold work. The hydrogen-occlusion behavior of the SAE 1020 is not compatible with the theory of occlusion by storage of molecular hydrogen.

To reconcile the various observations, it is proposed that two varieties of voids result from imperfect plastic flow of the ferrite about inclusions or other phases during cold rolling. Hydrogen may be stored in gaseous form in the larger of such voids, which do not disappear with annealing; but it is apparently associated in some other manner with narrower imperfections which are eliminated with annealing or recrystallization.

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Bend Plane Phenomena in the Deformation of Zinc Monocrystals

by John J. Gilman and T. A. Read

FOLLOWING the deformation of zinc monocrystals, sharply bent basal planes are observed near several types of inhomogeneities. Three of these inhomogeneities have characteristics which are quite regular so that they can be studied and analyzed. These are compressive kink bands, "deformation bands," and the inhomogeneities near end restraints. The present paper describes experiments in which "deformation bands" were artificially produced, and bend plane phenomena are discussed in terms of dislocation theory. Also, two new bend plane phenomena are described.

The importance of bend plane phenomena in the deformation of crystals is not widely recognized. Many phenomena may be explained in a manner similar to the discussion in this paper. Jillson[1] has pointed out that the "punching effect" in zinc is a bend plane phenomenon and is not caused by prismatic slip.[2] Bowles[3] has suggested that they may be involved in diffusionless phase changes. Cahn[4] has discussed the role of bend plane formation in the polygonization of zinc.

Experimental Work

Tensile Kink Bands: Because of the geometrical similarity between "deformation bands" and "kink bands" (compare Fig. 1 of this paper with Fig. 1 of the paper by Hess and Barrett[5]), the band shown in Fig. 1 of this paper will be called a "tensile kink band," and that shown by Hess and Barrett will be called a "compressive kink band." It is felt that the term "deformation band" should be reserved for banded structures in polycrystalline materials such as iron.[6]

Tensile kink bands seem to form spontaneously in aluminum crystals deformed by tensile loading.[7-10] In zinc and cadmium crystals they do not form in good, carefully loaded specimens.[1, 9] However, tensile kink bands can be produced artificially in zinc crystals. The present authors did this by scratching one of the flat surfaces of triangular crystals transversely with a sharp needle. Natural tensile kink bands caused by inhomogeneities sometimes appeared in deformed crystals which were identical in appearance with the artificially produced ones.

Zinc monocrystals were grown by the Bridgman method in graphite molds. Chemically pure zinc (99.999+ pct Zn) was used and the molds were sealed inside evacuated pyrex tubes during growth. The crystal cross sections were equilateral triangles with a typical base of 0.210 in. The artificial kink band shown in Fig. 1 is typical of tensile kink bands in zinc. The band lies between two bend planes which run from upper right to lower left and is inclined oppositely to the slip bands which are sharply bent at the two bend planes.

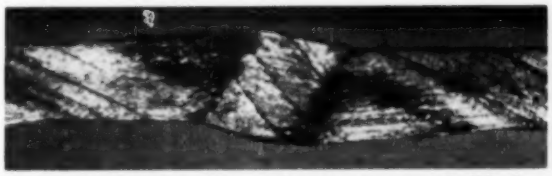

Fig. 1—Tensile kink band in a triangular zinc crystal. X4. Area reduced approximately 25 pct for reproduction.

The general form of the artificial tensile kink bands was independent of the scratch depth (1 to 5 mils deep) and also independent of which side was scratched. These variables did cause variations, however. Deep scratches produced more localized kink bands than light scratches. Also, if the angle between the slip plane traces and a transverse scratch varied appreciably among the three sides, then localization of the resulting kink bands also varied. Furthermore, if the slip direction lay nearly parallel to the scratched side, the band was more developed near the scratched side than at the opposite edge. Scratches produced tensile kink bands for crystal orientations from $\chi_o = 15°$ to $\chi_o = 75°$.

Fig. 2 shows a scratched crystal after deformation. One triangular side lies in the plane of the photograph. The right hand tensile kink band was produced by a transverse scratch on the upper right side. The next two kink bands were the result of scratches on the front surface. The kink band at the left was caused by a scratch on the lower back side. All four bands have the same general form.

A longitudinal scratch was also made on the crystal shown in Fig. 2 to determine the effect of a scratch on the critical shear stress. The critical shear stress of the scratched region was 33.9 g per sq mm compared to 24.4 g per sq mm for the unscratched region above it.

Fig. 3 shows Laue patterns of the crystal shown in Fig. 2. Fig. 3a shows the pattern of the undeformed crystal. The orientation was $\chi_o = 21°$, $\lambda_o = 31°$. After deformation, Fig. 3b was made of the homogeneously deformed portion of the crystal. The spots are compact but split into two halves. This region was elongated 45 pct and its orientation was $\chi = 14°$, $\lambda = 20°$; the sine law predicts $\chi = 14°$, $\lambda = 20.5°$. Fig. 3c was taken near the center of the middle tensile kink band of Fig. 2. The pattern shows a range of orientations and polygonization in this region. The spread in orientation was due to the fact that the basal planes were curved (see Fig. 1) rather than flat as in the ideal case. Some may also have been the result of elastic distortions and "local curvatures." The orientation range was $\chi = 23°$ to $32°$, $\lambda = 30°$ to $42°$. It is apparent from Fig. 3 that the material inside and outside the kink band rotated in opposite directions with respect to the tension axis during deformation. The orientation calculated from the ideal configuration of Fig. 9,

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This paper represents part of a thesis by J. J. Gilman submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy to the Graduate Faculties, Columbia University.

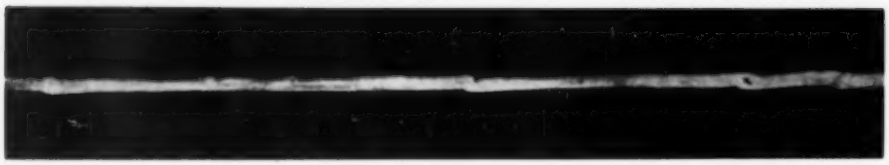

Fig. 2—Tensile kink bands produced in a triangular zinc crystal by scratches prior to elongation. X½. Area reduced approximately 20 pct for reproduction.

$\lambda_{bend} = 2\lambda_o - \lambda = 42°$, lies at the upper end of the observed range. Thus there is good agreement between the visual and X-ray observations.

To determine whether the effect of a scratch is localized or whether it extends far into a crystal, the following experiment was performed. A crystal was chemically polished and scratched transversely. Then about 0.25 mm of material was removed from each side of the specimen by means of chemical polishing. Another scratch was made on the same side of the crystal at a different place, and it was elongated in tension. An intense tensile kink band formed at the second scratch but only a faint one formed at the first scratch. The same result was obtained with a different crystal. Thus the effect of a scratch is localized initially and spreads across the diameter of a crystal as deformation proceeds.

Artificially produced tensile kink bands do not appear directly at the cross section which is scratched. They appear in the region of the undeformed crystal which is cut by a plane containing the line of the scratch and the normal to the basal planes of the crystal. This plane is one of the bend planes which bound the tensile kink band. Slip is initially inhibited only near the scratch, but after bend planes form the disturbance propagates across the width because of the self-stabilization of the kink band.

The authors have observed natural tensile kink bands near heterogeneities such as included crystals and nonmetallic inclusions. They also occur in mishandled crystals. Artificial tensile kink bands can be induced in zinc and tin crystals by scratches, but scratches have no effect for lead crystals. Schmid[11] showed that tensile kink bands can be induced in magnesium crystals by drilled holes. Both natural and artificial kink bands occur in variously shaped zinc crystals. In short, tensile kink bands tend to form whenever one part of a crystal lags in the reorientation process during a tensile test. Once formed, they are surprisingly stable against the large forces which tend to straighten them out. They are not observed when one portion of a crystal is softer than the rest. Two inhomogeneous regions analogous to those at end restraints are found at each end of the soft section instead. Kink bands do not form unless the hard part of the crystal is short compared to the diameter of the crystal. Thus no kink band formed at the longitudinally scratched region in Fig. 2, although it was harder than the rest of the crystal. If the hard section is long, the large reverse bending moments at the ends do not allow bend planes to form. Finally, tensile kink bands do not form when more than one slip system is active.[7]

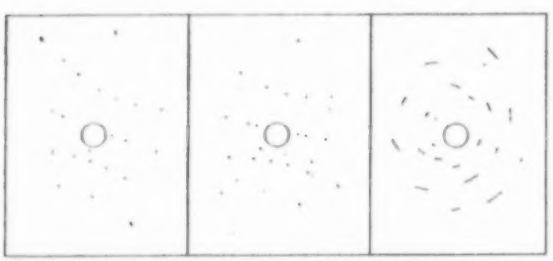

Fig. 3—Tracings of back-reflection Laue patterns. Tungsten radiation. a (left)—As-grown crystal. b (center)—After deformation, 45 pct elongation, homogeneously deformed portion. c (right)—After deformation, near center of deformation band.

New Bend Plane Phenomena: Two new bend plane phenomena have been observed. The first was observed when some crystals cleaved part way through their cross sections during elongation. Tongue-like pieces of the crystals separated from the rest and bent sharply backward upon further deformation, see Fig. 4. Some bending of such a tongue-like fragment would be expected even if the specimen were not a single crystal. The bending here is very localized, however. Also, the amount of bending was large compared to the additional deformation given the crystal after the partial cleavage formed. The very sharp angle between the outer edge of the bent piece and the edge of the crystal indicates a bend plane containing the sharp angle and bisecting the angle between the slip direction of the tongue and the slip direction of the rest of the crystal. If the specimen was deformed further, it is expected that the tongue would rotate oppositely to the slip planes in the rest of the crystal in order to maintain the symmetry across the bend plane.

Fig. 4—Bending of a piece of a partially cleaved crystal caused by deformation of the crystal subsequent to the partial cleavage. X2. Area reduced approximately 25 pct for reproduction.

The other new bend plane phenomenon was caused by a longitudinal scratch on one side of a triangular crystal (orientation was $\chi_o = 74°$, $\lambda_o = 74°$). Fig. 5a shows the side of the crystal opposite the scratch after deformation, about 50 pct elongation. Fig. 5b shows the cross section. Most of the cross section behaved normally during deformation. This is on the bottom in Fig. 5a. One corner, however, behaved abnormally; its slip planes rotated oppositely to the rest of the crystal. This is on the top in Fig. 5a. Thus, a sort of longitudinal kink band formed in contrast to the transverse kink band of Fig. 1. The anomalous corner did not coincide in location with the longitudinal scratch. If the two locations were connected by lines, these lines would lie near the basal plane normals. From this, and the fact that the specimen axis nearly bisects the angle between the slip directions of the two parts of the crystal, it is concluded that this phenomenon is similar to the effects of transverse scratches. It is believed that slip inhibition near the longitudinal scratch caused

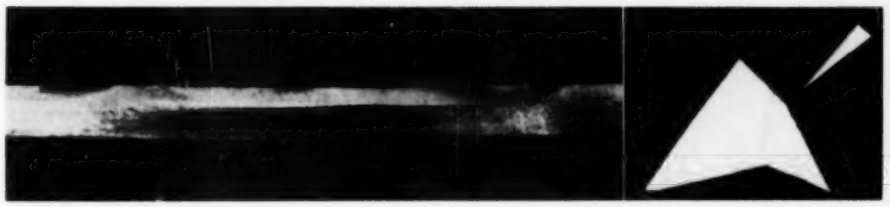

Fig. 5a (left)—Side of triangular crystal opposite longitudinal scratch (scratch on back upper side) after 55 pct deformation. X2. b (right) —Cross section of same crystal. Arrow points to scratched side. Area reduced approximately 25 pct for reproduction.

local bending when the crystal was elongated. This caused a longitudinal bend plane making a right angle with the slip direction. After the bend plane was established, the slip planes of the corner on the right in Fig. 5 rotated oppositely to the rest of the slip planes, leading to the configuration of Fig. 5.

Discussion and Theory

Tensile kink bands, compressive kink bands, and the bend planes at end restraints are closely related phenomena. The geometry of these will be discussed after a general dislocation theory of bend plane formation is presented.

Dislocation Theory of Bend Plane Formation: The role of dislocations in the formation of compressive kink bands was discussed by Hess and Barrett.[5] Their ideas can be extended by use of the dislocation theory of grain boundaries developed by Burgers[12] and by Read and Shockley.[13] First, it should be pointed out that it is not necessary to invoke dislocation concepts in order to explain bend plane formation phenomenologically. In zinc at room temperature, slip occurs only on the basal planes and therefore is lamellar. Because of this fact, a discontinuity in shear strain which results from slip must lie in a plane which bisects the disorientation angle between the lattices on either side of the discontinuity. If the shear discontinuity does not lie in the bisecting plane, as shown in Fig. 6a, the thicknesses of the sheared lamellae are reduced; an impossible circumstance. Fig. 6b shows that when a bend plane forms, the strain in the sheared material is given by 2 tan $\theta/2$, where θ is the disorientation angle.

In terms of dislocation theory, bend planes consist of walls of dislocations having the same sign; that is, they are grain boundaries. Defined in physical terms, they are also grain boundaries but are usually discussed as distinct phenomena because they have a different origin than ordinary grain boundaries. Bend planes are rather ideal grain boundaries in that they have only one degree of freedom; the angle between the slip directions on either side of the bend plane.

Bend planes in bent crystals decrease the energy of the system. A plastically bent zinc crystal contains an excess of positive dislocations if it is bent concavely downward. The density of excess positive dislocations is inversely proportional to the radius of curvature.[4] These excess positive dislocations may be considered to constitute a multitude of small angle grain boundaries. The energy of this configuration may be compared with that of a bent crystal containing a single large-angle grain boundary.

The Read and Shockley expression for the energy of a grain boundary is

$$E = E_o \theta \, (A - \ln \theta)$$

where A and E_o are constants and θ is the disorientation angle. The boundary bisects θ. If ϕ is the angle between the two ends of a bent specimen and it contains $M + 1$ grains, hence M grain boundaries, the orientation difference across each boundary is ϕ/M. Thus the total grain boundary energy of a specimen with variable M will be

$$E = E_o \phi \, [A - \ln(\phi/M)]$$

Since ϕ/M must always be small if the theory is to be valid, this expression increases continuously with increasing M. Thus the energy is lowest if the orientation difference between the ends is contained in one boundary. However, if ϕ/M is large, and hence the theory only approximate, there may exist values of ϕ for which the energy will be lower if two twin boundaries are present instead of one large-angle boundary. The bend plane always bisects the angle between the lattices on either side of it. Also, the bend plane contains the normal to the plane containing the slip directions. This position is stable because it yields a maximum of symmetry across the bend plane and a minimum dislocation density in the bend plane. This can be verified by the method of Frank.[14]

Although a bent specimen having one large-angle grain boundary has a lower energy than one with many slightly disoriented grains, bend planes do not always form spontaneously in bent zinc crystals. For example, Cahn[4] found that uniformly bent zinc crystals do not polygonize unless they are heated for several hours near the melting point. The temperature necessary to cause polygonization is lower the smaller the bend radius, and the longer the annealing time. Similar results were found for silicon ferrite by Dunn and Daniels.[15] On the other hand, bend

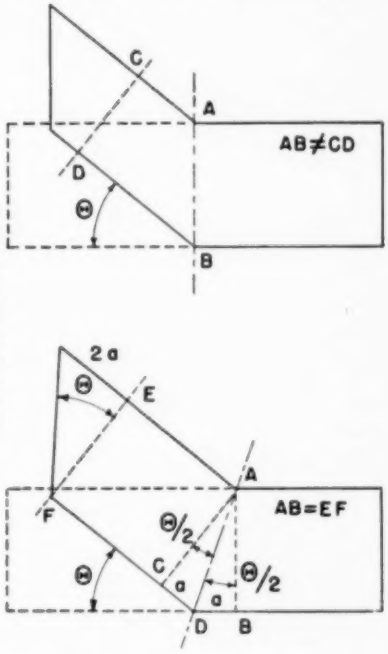

Fig. 6—Showing that the lamellar nature of deformation by slip requires that a shear discontinuity lie in a plane which bisects the disorientation angle between the lattices on either side of the discontinuity.

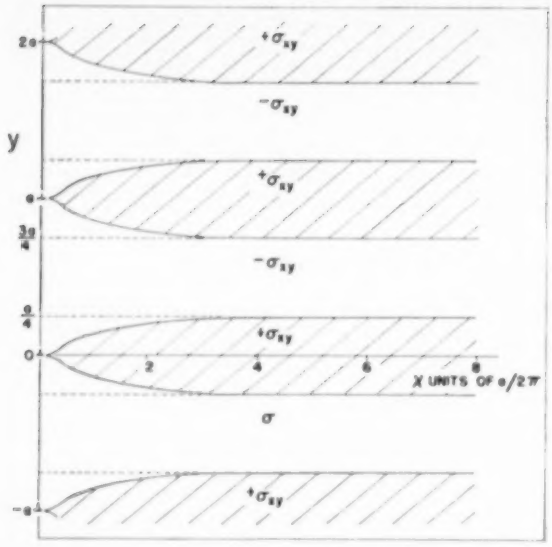

Fig. 7a—Shear stresses caused by an infinite vertical wall of positive edge dislocations. From Burgers' Eq. 13. Regions of positive and negative shear stress over the xy plane.

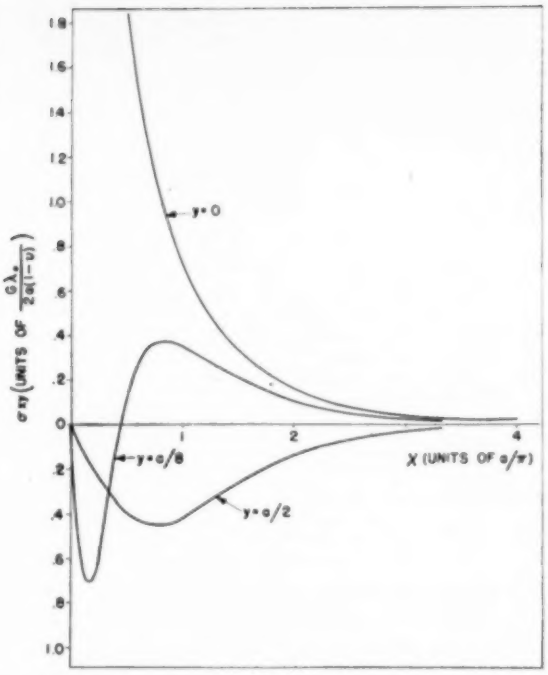

Fig. 7b—Shear stresses caused by an infinite vertical wall of positive edge dislocations. From Burgers' Eq. 13. Variation of shear stresses with x at various values of y.

planes form at room temperature at kink bands and the Laue pattern of Fig. 3c shows evidence of partial polygonization within a tensile kink band.

In a uniformly bent zinc crystal it is presumed that the excess positive dislocations are rather uniformly distributed in a rectangular array. Because of the mutual repulsive forces between them, they tend to remain so distributed. At high temperatures, because of thermally actuated fluctuations, they gradually join into vertical arrays and thus polygonize the bent crystal. If the initial bending is nonuniform, as it is for bend plane formation, the excess positive dislocation density on a given plane is greatest at the point of greatest bending. This configuration is stable and can attract other dislocations to itself. Consideration of an expression which Burgers[12] derived will clarify this statement.

Burgers' expression for the shear stress due to an infinite vertical wall of uniformly spaced positive edge-dislocations is

$$\sigma_{xy} = \left[\frac{G\,l}{2\pi(1-\nu)}\right]\frac{\pi^2 x}{a^2}\left[\frac{\cos h\left(\frac{2\pi x}{a}\right)\cos\left(\frac{2\pi y}{a}\right) - 1}{2\left[\sin h^2\left(\frac{\pi x}{a}\right) + \sin^2\left(\frac{\pi y}{a}\right)\right]^2}\right]$$

where x, y are Cartesian coordinates; a, the spacing of the dislocations in the wall; G, the shear modulus; l, the magnitude of the slip vector of the dislocations; and ν is Poisson's ratio. The boundaries of regions of positive and negative shear stress in the xy plane are found by setting the numerator of the equation equal to zero, that is, by plotting

$$\cos h\left(\frac{2\pi x}{a}\right)\cos\left(\frac{2\pi y}{a}\right) = 1$$

This has been done in Fig. 7a. At large x the shear stress varies sinusoidally with y. It is positive in the shaded regions from $-a/4$ to $+a/4$, from $3a/4$ to $5a/4$, etc. Therefore a positive dislocation at large x within those regions would be repulsed from the wall. On the other hand the shear stress is negative in the regions $a/4$ to $3a/4$, $5a/4$ to $7a/4$, etc., and dislocations within those bands would be attracted to the wall. At small x the bands of positive shear stress become very narrow. Therefore a dislocation at small x and almost any y would be attracted to the wall.

The variation of the shear stress, σ_{xy}, with x for various values of y, is plotted in Fig. 7b. For $y = 0$ the shear stress is positive for all x and approaches infinity as $x \to 0$. For $y = a/8$ the shear is negative for small x but positive for large x.

Since the positive dislocations are inhibited from entering the wall if they lie in planes near those in which the wall already has dislocations, the wall spacing tends to be uniform. Also, a wall of positive edge dislocations is self-stabilizing. As dislocations are added to the wall, a decreases so shear stresses due to the wall become increasingly localized.

All of the bend plane phenomena which have been discussed in this paper began with nonuniform bending of specimens. It is believed that this caused a few excess dislocations of the same sign to assemble in the planes of maximum bending. Thus vertical walls were formed which became increasingly stable as more dislocations attached themselves. The actual experimental configurations are non-ideal, of course. Instead of single dislocations perhaps bend planes may be thought of as assemblies of dislocation bunches. These would behave approximately as single dislocations having large slip vectors.

Bend Planes at End Restraints: Bend planes at end restraints were first described by Mark, Polanyi, and Schmid.[10] Later, Miller[17] studied them systematically. Fig. 8a is taken from Miller's paper. It shows that, at the restrained end of a crystal, the

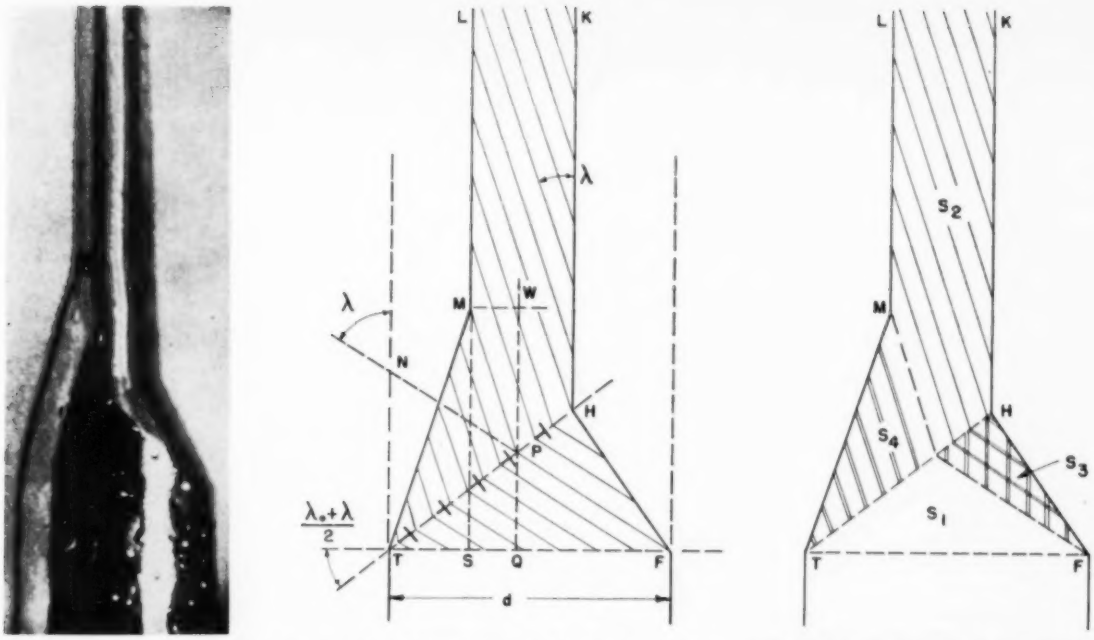

Fig. 8—Bend planes at end restraints. a (left)—Inhomogeneous deformation in a zinc monocrystal near end restrained by polycrystalline portion of rod. (After Miller.) X3. b (center)—Schematic geometry of Fig. 8a. c (right)—Homogeneous shear strain regions in Fig. 8a.

basal planes are bent during deformation. The bending is about an axis lying in the slip plane and normal to the slip direction. The bending moment reaches a maximum at the plane *TF*, Fig. 8b, but the maximum amount of plastic bending occurs at a plane which is normal to the slip direction and passes through point *T* because the material within *TPF* is restrained from slipping by the end restraint. Excess dislocations of the sign shown assemble along *TH* and form a dislocation wall. Once it is formed, the wall is stable against dispersion and maintains a position such that it bisects the angle *MPF* between the slip directions of the lattice on either side of it. In order to do this, *TH* is required to pivot about *T*, because the planes within *TPF* cannot easily change in orientation. This pivoting is a rather ideal case of stress-induced, grain-boundary migration.

Actual crystals approach the ideal behavior depicted in Fig. 1b more closely at elevated temperatures than at room temperature.[2] This happens because recovery (annihilation of dislocations) is rapid at elevated temperatures so that not many excess dislocations pile up adjacent to the bend plane and cause distortion. Miller showed, experimentally, that the bend plane *TH* always made an angle with *TF* of $\frac{1}{2}(\lambda_o + \lambda)$, where λ_o is the initial angle between slip direction and tension axis. Thus, the position of *TH* can be calculated for any tensile elongation from the laws of crystallographic slip.[18] Also, it can be seen that the bend plane *TH* always bisects the angle between the initial and final slip directions, as seen from the geometry of Fig. 6b. The analysis which Miller carried out was incomplete because he did not calculate the maximum distance at which the end restraint affects the crystal, *MS*. Also, he did not calculate the shear strains in the various regions of the specimen.

The distance *MS* can be calculated. This derivation was suggested by W. T. Read in a private communication. The suggestion of McKeehan in his discussion of Miller's paper cannot be used to calculate *MS* because the axes of the deformed and undeformed sections do not coincide. The calculation is

$$MP = NF - PF$$
$$= d \csc \lambda_o - PQ \sec \lambda_o$$

and

$$MS = MP \cos \lambda + PQ$$

Miller showed that

$$PQ = d \left[\tan \lambda_o + \cot \left(\frac{\lambda + \lambda_o}{2} \right) \right]$$

Therefore,

$$\frac{MS}{d} = \frac{\cos \lambda}{\sin \lambda_o} + \frac{1 - \cos \lambda \sec \lambda_o}{\tan \lambda_o + \cot \left(\frac{\lambda + \lambda_o}{2} \right)}$$

With manipulation this reduces to

$$\frac{MS}{d} = \frac{\cos \lambda}{\sin \lambda_o} - \sin (\lambda_o - \lambda) \left[\frac{1 - \cos (\lambda_o + \lambda)}{1 + \cos (\lambda_o - \lambda)} \right]$$

MS was calculated for Miller's data by means of the last expression and the calculated values agreed satisfactorily with the observed values.

The four regions of homogeneous shear near an end restraint are shown in Fig. 8c. In the region *TPF* the shear strain S_1 is equal to zero. Consideration of Fig. 6b shows that in the region *TMP* the shear strain is

$$S_4 = 2 \tan \left(\frac{\lambda_o - \lambda}{2} \right)$$

The standard expression for the shear strain in the region *LMPHK*[19] is

$$S_2 = \cot \lambda - \cot \lambda_o$$

Since the shear strain discontinuity across *TH* is constant, then

$$S_2 - S_3 = S_4 - S_1$$

Hence

$$S_3 = S_2 - S_4$$

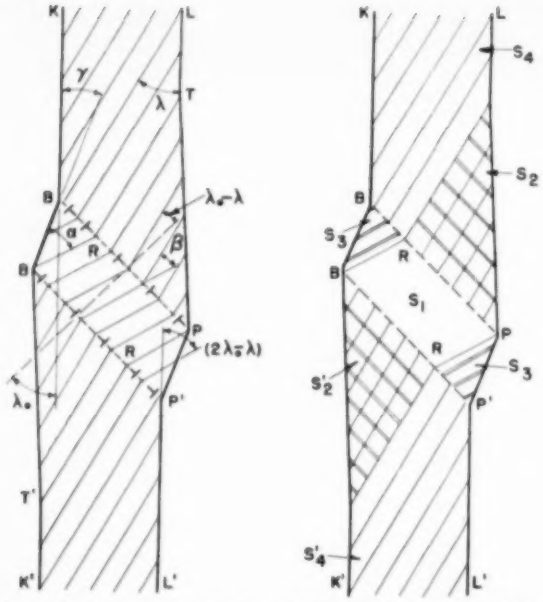

Fig. 9—Schematic drawings of an ideal tensile kink band. a (left) — Schematic geometry. b (right) — Regions of differing but homogeneous shear strain.

Thus the configuration near an ideal end restraint may be completely analyzed.

Tensile Kink Bands: Tensile kink bands in zinc crystals were also first studied systematically by Miller.[10] His interpretation of them was incorrect, however, because he thought they were caused by a new form of twinning. Later authors, notably Andrade[20] and Jillson,[1] corrected this mistaken impression, but did not discuss the bands in detail.

If a localized portion of a crystal is harder than the rest, bending moments are created at the edges of the hard portion when the adjacent soft portions slip. The resulting excess dislocation concentrations result in dislocation walls which form along planes BP and $B'P'$, Fig. 9a, cutting the slip planes at right angles to the slip direction. These walls stabilize and tend to maintain equal angles between themselves and the slip directions on either side. As slip causes the basal planes outside the band to rotate counterclockwise, the dislocation walls at BP and $B'P'$ cause the basal planes within the band to rotate equally clockwise. When the bend planes become saturated with dislocations, dislocations of the same sign begin to pile up outside. These cause curvature of the basal planes near the bend planes to give the non-ideal configuration of Fig. 1.

The drawing of Fig. 9a shows the schematic geometry of an ideal tensile kink band. The angle between the slip planes within the kink band and the tension axis is $(2\lambda_o - \lambda)$ where λ_o and λ are the initial and final angles between the slip direction and the tension axis respectively.

It should be noted that the rotation of the slip planes within $BPP'B'$ may occur either by means of reverse slip in $BPP'B'$ or by a bulk rotation of the region combined with lateral movement of the bend planes (rotation of the bend planes may occur but it causes no rotation in the region $BPP'B'$). Since no experimental evidence of reverse slip was found, it is believed that the latter mechanism operates when an ideal tensile kink band forms. The rotation of the planes relative to the sides BB' and PP' is the result of normal forward slip in $B'BR$ and $PP'R'$. This latter rotation causes the slip planes within a kink band to take the angle α with the side BB' and the side BB' takes an angle γ with the tension axis. If the angle α is known, γ may be calculated as follows:

$$\gamma = \beta - (\alpha - \lambda_o)$$
$$= 3\lambda_o - (\alpha + 2\lambda)$$

Fig. 9b shows the various regions of homogeneous shear strain. The shear strains are

$$S_1 = 0$$
$$S_2 = S_2' = 2 \tan \beta/2$$
$$S_3 = S_3' = \cot \alpha - \cot \lambda_o$$
$$S_4 = S_4' = \cot \lambda - \cot \lambda_o$$

Since the shear strain discontinuities across BP and $B'P'$ are constant, then

$$S_x = S_4 - S_3 = S_2' = S_4' - S_3'$$

Thus an ideal tensile kink band is completely determined except for thickness if the angle α, as well as λ_o and λ, is known.

Actual tensile kink bands deviate from the ideal case because some slip occurs in the region $BPP'B'$ after the band has formed. This was shown by forming a kink band in a specimen, then electropolishing to remove the slip lines, and finally elongating the specimen an additional 10 pct. Slip lines could be seen within the kink band, at the bend planes, and outside the kink band where the deformation was homogeneous. This means that the planes BP and $B'P'$ do not represent static discontinuities since some material passes through them.

Compressive Kink Bands: Compressive kink bands were discovered by Orowan[21] in cadmium monocrystals which had very low orientation angles. He believed that an entirely new mechanism of deformation was involved, but Hess and Barrett[1] were able to form compressive kink bands slowly in zinc and thought that the mechanisms of slip and bend plane formation explained their observations. Compressive kink bands differ in their mode of formation from tensile kink bands. Hess and Barrett[1] showed that buckling is necessary for compressive kink-band formation. This buckling must be that of a beam with restrained ends. If the ends are not restrained, the crystal simply bends uniformly. This is consistent with the theory discussed above. It is expected that a crystal bent with free ends would polygonize if annealed, but bend planes would not form in it during deformation. If the ends of the crystal are restrained, the crystal buckles with two points of inflection in the displacement curve at which the shear stresses are a maximum. Shear yielding occurs here first, according to Hess and Barrett. Thus, positive and negative dislocations flow out of this region along the length of the crystal (the basal planes in this case must be almost parallel to the compression axis) toward the points of maximum bending and minimum shear stress. They stop somewhere in between and assemble into dislocation walls. These oppositely directed walls or bend planes constitute the initial stage in the formation of a compressive kink band.

An important distinction between compressive and tensile kink bands is that the former can be formed in an otherwise homogeneous zinc crystal while the latter form only in crystals containing heterogeneities. Compressive kink bands form in a

particular type of test (compression of thin rod with restrained ends) in crystals having rather restricted orientations. Tensile kink bands form in a particular test (axial tension of thin rod) in crystals with localized heterogeneities. Thus, neither phenomenon is a fundamental mode of deformation. They are the result of the interactions between particular mechanical tests and particular crystals. Their general interest derives from the fact that they involve the formation of bend planes and these seem to be important to our understanding of the behavior of dislocations.

Summary

The inhomogeneities of deformation which occur at the restrained ends of zinc crystals, at tensile kink bands, and at compressive kink bands are described in terms of bend planes which are believed to consist of walls of edge dislocations. The bend plane bisects the angle between the slip directions on either side of it and contains the normal of the plane of the two slip directions. This is used to explain the migration of the bend plane through the lattice at an end restraint, the "anomalous" rotation of the lattice within a tensile kink band, and the geometric conditions which obtain during the formation of a compressive kink band.

Experiments are described in which artificial tensile kink bands were formed by transverse scratches. Scratches cause tensile kink bands in zinc crystals of a range of orientations; which side is scratched is relatively unimportant. It is concluded that tensile kink bands form in zinc if a specimen contains a region of inhibited slip. The lag of the inhibited slip planes in the reorientation process causes bending moments on either side of the inhibited planes. These lead to bend plane formation and thence to a tensile kink band.

Two new bend plane phenomena are described. One of these results when a partially cleaved crystal is elongated. The tongue-like projection resulting from the partial cleavage bends sharply away from the rest of the crystal. This phenomenon is explained by the formation of a bend plane at the base of the projection. The other new phenomenon is caused by a longitudinal scratch on a zinc crystal. Under certain geometrical conditions this causes a bend plane to form parallel to the tension axis of the crystal.

Acknowledgment

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Technical Note

A Replica Method for Study of the Structure of Lead-Antimony Alloys

by Jeanne Burbank

A TECHNIQUE has been developed for the microscopic study of the three-dimensional structure of the Pb-Sb alloys by the formation of a chemical replica in which the internal structure of the metal may be seen directly. In the replica process one of the phases present in the structure as-cast is converted into a translucent material, and the second phase remains virtually unaffected, permitting microscopic examination by transmitted light.

Alloys of lead and antimony were made from the chemically pure metals, cast in a sooted and pre-

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Fig. 1—Chemical replica of 4 pct Pb-Sb alloy. X160. Area reduced approximately 50 pct for reproduction.

Fig. 2—Chemical replica of 12 pct Pb-Sb alloy showing a crystal of primary antimony. X2000. Area reduced approximately 50 pct for reproduction.

heated brass mold, then air cooled. A vacuum pump was used to draw the molten metal into the mold, producing a sheet 0.003 to 0.004 in. thick. By this technique foils were cast having microstructures similar to those in massive castings except that in most cases the foils were one grain thick. A smooth finish is required on the mold faces to assure ready release of the cast foil and because the specimens may not be polished after casting, owing to their thinness and softness.

The replicas were prepared by prolonged treatment of the as-cast foil specimens with the familiar acetic acid-superoxol etchant.[1] The metal foil, supported on a microscope slide or cover glass, was placed in a shallow dish containing etchant. After a period of several hours the foil was converted to a replica and removed to a slide for microscopic examination. The replica was illuminated by transmitted light and micrographs made. The replicas were also examined with the stereoscopic microscope, which clearly showed the three dimensional distribution of the two phases. Chemical replicas were thus prepared and examined for 1, 4, 9½, and 12 pct Pb-Sb alloys. The replicas are fragile and must be handled with care. It has also been found necessary to make all examinations while specimens are still moist because the translucent material becomes powdery and opaque upon drying.

As evidenced by X-ray diffraction determinations, the replicas are composed of amorphous material and metallic antimony. The white amorphous material contains lead, also trivalent and pentavalent antimony.[2] This is possibly a mixture of lead metantimonate with tetroxide and trioxide of antimony. The opaque material, which appears black in the accompanying micrographs, is identified by X-ray diffraction as metallic antimony.

Fig. 1 is a chemical replica of a 4 pct Pb-Sb alloy at low magnification showing the dendritic formations present in the cast metal. The translucent material is found in those areas originally occupied by the lead solid solution. The black portions are the unattacked antimony-rich phase segregated in the interdendritic and intergranular areas. It is evident from this projection of the three-dimensional structure that the entire structure is dendritic, but in many cases the antimony is distributed across dendrite arms; the arms lie at random angles to the specimen surface; and the primary, secondary, and tertiary branches are of varying sizes. For these reasons a metallographic etch of a random cut through such a specimen will often fail to show any recognizable dendritic formation.

At low magnifications, the replica structure of the hypoeutectic alloys resembles that shown in Fig. 1. Examination at higher powers shows that segregated antimony is spheroidal in the low alloys up to approximately 6 pct Sb, and above this concentration the segregated antimony assumes more definite shapes, resembling the eutectic structure commonly seen in metallography. Primary crystals of antimony appear as the eutectic point is exceeded, as in Fig. 2, from a replica of a 12 pct Pb-Sb alloy.

It is intended to use the replica technique to investigate the effects of heat treatment, working, and aging of Pb-Sb alloys. For example, homogenized alloys should produce uniform translucent replicas containing no particles of segregated antimony; annealed specimens should show agglomeration of the antimony into discrete crystals, and the accompanying changes in the lead dendrites; Widmannstätten structure, which is difficult to demonstrate metallographically in these alloys[3] should be rendered readily visible in the replicas.

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Preferred Orientations in Iodide Titanium

by Carl J. McHargue and Joseph P. Hammond

The wire textures for cold rolled and recrystallized iodide titanium and the sheet textures for this material produced by cold and hot rolling, and recrystallization at a series of temperatures were determined. The effect of the $\alpha \rightarrow \beta$ transformation on the sheet texture was noted.

UNTIL recently it was believed that all hexagonal close-packed metals deformed by slip on the basal plane, (0001), and that rolling should tend to rotate this slip plane into the plane of the rolled sheet. The pole figures of cold rolled magnesium[1] are satisfactorily explained on this basis. There is a tendency for the $<11\bar{2}0>$ directions to align parallel to the rolling direction, and the principal scatter is in the rolling direction. Zinc[2] has a rolling texture in which the hexagonal axis is inclined 20° to 25° toward the rolling direction. Twinning is believed to account for the moving of the basal plane away from parallelism with the rolling plane. The texture of beryllium[3] places the basal plane parallel to the rolling plane with the $[10\bar{1}0]$ direction parallel to the rolling direction, and the scatter from this orientation is primarily in the transverse direction. Cold rolled textures reported for zirconium[4] and titanium[5] show the $[10\bar{1}0]$ directions to lie parallel to the rolling direction and the (0001) plane tilted by approximately 25° to 30° to the rolling plane in the transverse direction.

Rosi[6] has recently reported that the mechanisms for deformation in titanium are distinctly different from those commonly reported for hexagonal close-packed metals. The principal slip plane is the prismatic plane, $\{10\bar{1}0\}$, with some slip also occurring on the pyramidal planes, $\{10\bar{1}1\}$. However, there is no evidence for basal slip. The slip direction is reported to be the close-packed digonal axis, $[11\bar{2}0]$. In addition to the twin plane commonly reported for metals of this class, $\{10\bar{1}2\}$, Rosi found the twin planes $\{11\bar{2}2\}$ and $\{11\bar{2}1\}$, with the dominant twin plane being $\{11\bar{2}1\}$.

Information regarding the recrystallization and hot rolling textures of hexagonal close-packed metals is limited. Barrett and Smigelskas report that rolling beryllium at temperatures up to 800°C and recrystallization at 700°C produce textures not differing from the cold rolled sheet texture.[3] McGeary and Lustman find that hot rolling at 850°C produces the same basic texture in zirconium as rolling at room temperature.[4] These investigators also report that the texture for sheet zirconium recrystallized at 650°C differs from the cold rolled orientation inasmuch as the $[11\bar{2}0]$ direction, instead of the $[10\bar{1}0]$ direction, is parallel to the rolling direction. In the case of titanium, it is not possible to deduce which direction is preferred in the recrystallized state from the pole figures presented by Clark.[5]

The purpose of this paper is to report an extensive investigation of the preferred orientations in iodide titanium. Since the deformation mechanisms for titanium are different from those commonly given for hexagonal close-packed metals, it is not surprising to find distinct differences between the textures of titanium and other metals of this class.

Materials and Methods

This investigation was carried out on iodide titanium obtained from the New Jersey Zinc Co. with an analysis as follows: N_2, 0.002 pct; Mn, 0.004; Fe, 0.0065; Al, 0.0065; Pb, 0.0025; Cu, 0.01; Sn, 0.002; and Ti, remainder.

The crystallities of titanium were broken from the as-deposited bar and melted to form 20 g buttons on a water-cooled copper block in a vacuum arc-furnace. Hardness tests conducted on the material before and after melting differed by only two or three Vickers Pyramid Numbers, indicating no or insignificant contamination. The buttons were hot forged, ground, and etched to sizes and shapes suitable for the rolling schedule, and vacuum annealed at 1300°F. Specimens for determination of the wire textures were reduced 91 pct in diameter to 0.027 in. in 24 steps using grooved rolls. In order for the orientation of the central region to be studied, portions of these wires were electrolytically reduced to a diameter of 0.005 in. using the procedure described by Sutcliffe and Reynolds.[7] The sheet textures were determined on titanium cold rolled 97 pct to a thickness of 0.005 in. A reduction of approximately 10 pct per pass was used, and the rolling direction was changed 180° after each pass.

Specimens used for determination of the recrystallized textures were annealed in evacuated quartz tubes at 1000°, 1300°, and 1500°F. The grain size of the 1000°F specimen was sufficiently small to give satisfactory X-ray patterns with the specimen stationary. However, it was necessary to scan the surface of the other recrystallized specimens. The microstructure of each annealed specimen was that of a recrystallized material. The diffraction rings all showed the break-up into spots typical of recrystallized structures.

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This paper represents part of a thesis by C. J. McHargue submitted in partial fulfillment of requirements for degree of Doctor of Engineering to the Graduate School, University of Kentucky.

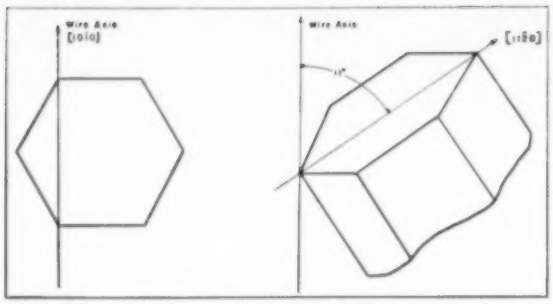


Fig. 1—Fiber axis in iodide titanium.

In order that the orientations in titanium deformed at elevated temperatures could be investigated, annealed ingots were reduced 95 pct in thickness to 0.010 in. at 1050° and at 1450°F. The specimens were heated in air and rolled between sheets of mica to minimize heat losses. The material was reheated after each reduction, which amounted to approximately 10 pct of the sheet thickness. These specimens were etched slightly to remove the surface contamination.

The effect of the $\alpha \rightarrow \beta$ transformation on the preferred orientation was found for a specimen cold rolled 97 pct, heated 5 min at 1650°F in vacuum, and very slowly cooled.

The X-ray specimens were made in the shapes of small rods by polishing strips in a specially constructed jig and etching away the material affected by this polishing. The data for samples of larger grain sizes were obtained by shots on sheet material in a structure integrating camera.[9] Transmission photograms were taken with molybdenum radiation (45 kv, 18 ma) and a 0.030 in. pinhole. Since titanium fluoresces under these conditions, it was necessary to use a zirconium filter between the specimen and film. Exposures were made with the beam perpendicular to the rolling direction at 0, 11°, 26°, 41°, 56°, 71°, and 79° to the cross direction, and with the beam perpendicular to the cross direction at 11°, 26°, 56°, 71°, and 79° to the rolling direction. Additional exposures were then made where necessary. The variations in intensity of the diffraction rings were estimated by eye with the aid of an exposure chart. Intensity readings of 0, 1, 2, and 3 were made on all specimens except those hot rolled, for which intensities of 0, 1, and 2 were used.

Discussion of Results

Wire Textures: The orientation of the cold rolled wire can be described as having the [10$\bar{1}$0] direction, or type II digonal axis, parallel to the wire axis with all azimuthal position possible. This is shown schematically in Fig. 1a. Burgers, Fast, and Jacobs report the same wire texture for zirconium,[9] and Morell and Hanawalt report it for Dowmetal.[10]

The texture at the surface and the center is essentially the same, varying only in the degree of scatter about the ideal. From a measurement of the spread of the diffraction spots,[11] it was found that the [10$\bar{1}$0] direction has a spread of ±6° to the wire axis in the central region and ±15° in the surface layers.

The maxima of the diffraction rings for the recrystallized specimens were found to be split and joined by regions of lesser intensity. The average values of the maxima for the (0002), (10$\bar{1}$0), (10$\bar{1}$1), and (11$\bar{2}$0) planes give the average orientation as having the [11$\bar{2}$0] direction, or type I digonal axis, parallel to the wire axis. However, this orientation is accurately described as having strong concentrations of the basal planes tilted ±11° to the wire axis with some basal planes at all positions between these. The [11$\bar{2}$0] direction is located so that it would be parallel to the wire axis if the basal plane

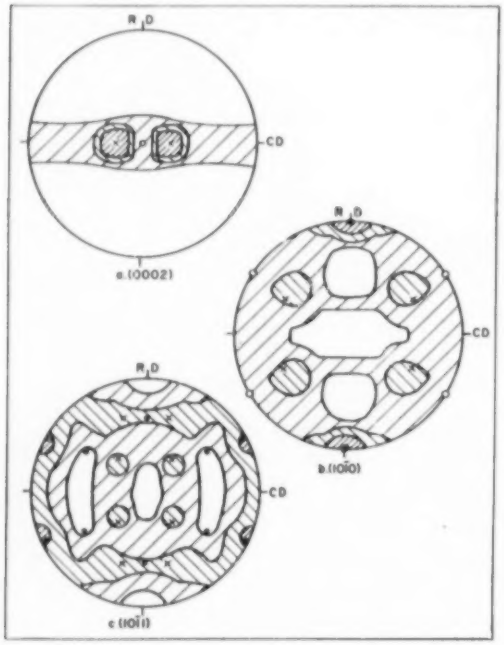


Fig. 2—Cold rolled sheet texture of iodide titanium. o = (0002) [10$\bar{1}$0]. x = (0002) rotated 27° about rolling direction.

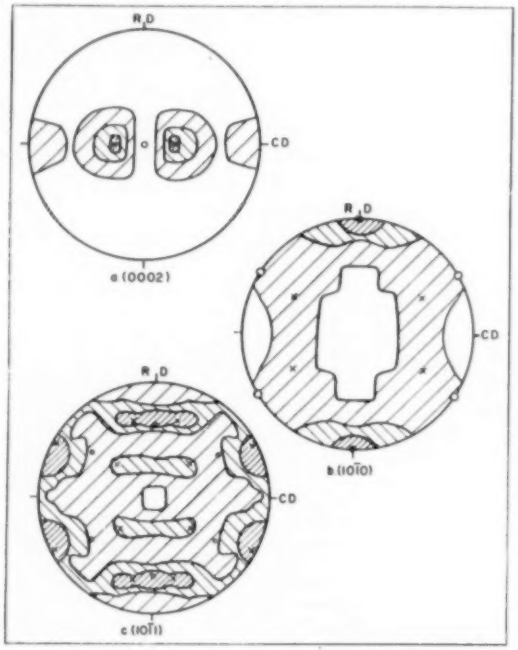


Fig. 3—Cold rolled annealed texture of iodide titanium. o = (0002) [10$\bar{1}$0]. x = (0002) rotated 27° about rolling direction.

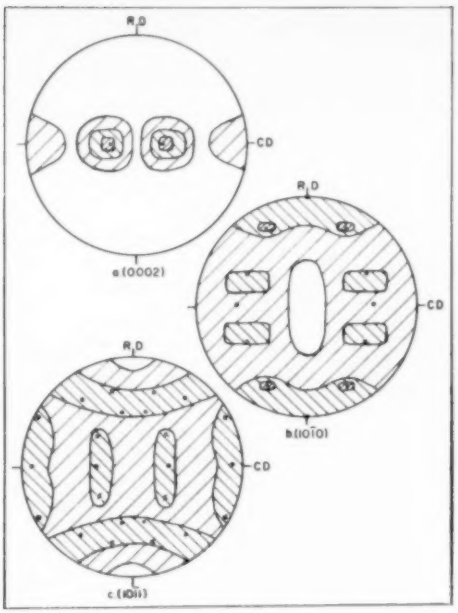
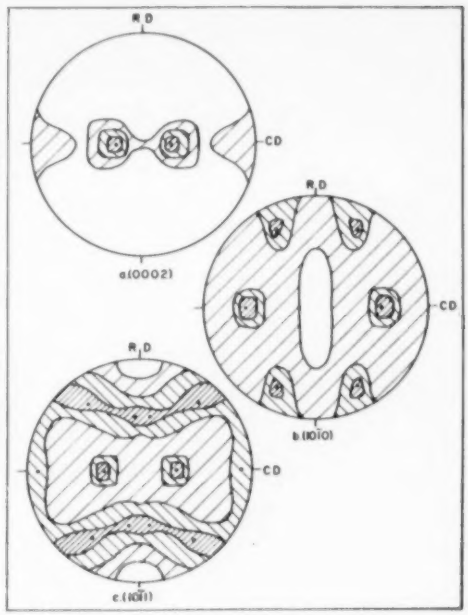


Fig. 4 (left) — Iodide titanium cold rolled 97 pct, annealed at 1300°F. • = (0002) rotated 27°, [11$\bar{2}$0] parallel to rolling direction. x = (0002) rotated 27°, [10$\bar{1}$0] parallel to rolling direction.

Fig. 5 (right) — Iodide titanium cold rolled 97 pct, annealed at 1500°F. • = (0002) rotated 27°, [11$\bar{2}$0] parallel to rolling direction.



were to be rotated until parallel to the wire axis. The orientation is such that the (21$\bar{3}$0) plane is normal to the wire axis, which is shown schematically in Fig. 1b.

Exposures on the surface layer do not show the strong, split maxima, but indicate the average position of [11$\bar{2}$0] to lie along the wire axis with a spread of ±35°. The recrystallized wire texture of zirconium[3] has been found to be [11$\bar{2}$0].

Cold Rolled Sheet Texture: The cold rolled sheet texture for iodide titanium is presented in Fig. 2. Fig. 2a shows that two high intensity regions are developed for (0002) planes, which correspond to rotation of approximately 27° in the transverse direction from the "ideal" orientation of (0002) [10$\bar{1}$0] reported for other hexagonal metals. McGeary and Lustman found indications of this tilt in zirconium,[4] and it has been reported in titanium by Clark.[5] Fig. 2b shows that the [10$\bar{1}$0] direction is aligning parallel to the rolling direction, and Fig. 2c confirms the selection of (0002) rotated 27° and [10$\bar{1}$0] parallel to the rolling direction as the orientation describing the texture. Most of the spread from this "ideal" orientation is in the transverse direction.

Cold Rolled Recrystallized Sheet Texture: Fig. 3 presents the pole figures representing the orientation of the material annealed at 1000°F. These pole figures show that the cold rolled texture is retained for recrystallization at a relatively low temperature, and that there is a slight sharpening of the texture.

An investigation of material cold rolled 97 pct, and vacuum annealed at 1300°F indicates that a dual texture is present in this condition. As is shown in Fig. 4, this texture can be explained in terms of the tilted basal plane and one component having the [10$\bar{1}$0] direction parallel to the rolling plane, and another component having the [1120] direction parallel to the rolling direction.

The pole figures for iodide titanium cold reduced 97 pct and annealed at 1500°F, Fig. 5, are describable in terms of the basal plane tilted approximately 27° and the [11$\bar{2}$0] direction aligned with the rolling direction.

Thus, it is seen that the texture obtained upon recrystallization depends upon the temperature of treatment. This work indicates that the cold rolled texture is retained by the material just recrystallized. However, as the temperature is increased and grain growth and secondary recrystallization take place, a new texture is produced.

The texture reported for zirconium recrystallized at 1200°F differs from the cold rolled texture by a rotation of 30° about the normal to the basal plane.[4] Beck[12] and Kronberg and Wilson[13] point out that there is a component of the recrystallized textures of face-centered cubic metals which corresponds to a rotation of 30° to 40° about a [111] axis. The results of the present investigation lend added support to Beck's conclusion that in hexagonal close-packed and face-centered cubic metals, the reorientation upon recrystallization corresponds to a rotation about the normal of a close-packed plane.

Hot Rolled Texture at 1050°F: With the exception of beryllium[3] and zirconium,[4] there is no information on the preferred orientation produced by hot rolling hexagonal close-packed metals. These two materials retain their cold rolled textures when rolled at temperatures as high as 1475°F.

The pole figures for titanium hot rolled at 1050°F, Fig. 6, show that this texture can satisfactorily be described in terms of the "ideal" orientation (0002) [10$\bar{1}$0]. Fig. 6a shows a tendency for the (0002) planes to be tilted as in the cold rolled sheet texture, and Fig. 6b shows the tendency for the [10$\bar{1}$0] direction to be aligned parallel to the rolling direction. The spread of the (0002) plane in the cross direction is ±40°. The scatter about the normal to the rolling plane brings the [11$\bar{2}$0] directions parallel to the rolling direction at the ends of this spread. Again, the spread is greater in the transverse direction than in the rolling direction.

That the hot rolled texture is describable in terms of the orientation (0002) [10$\bar{1}$0] is shown by Fig. 6.

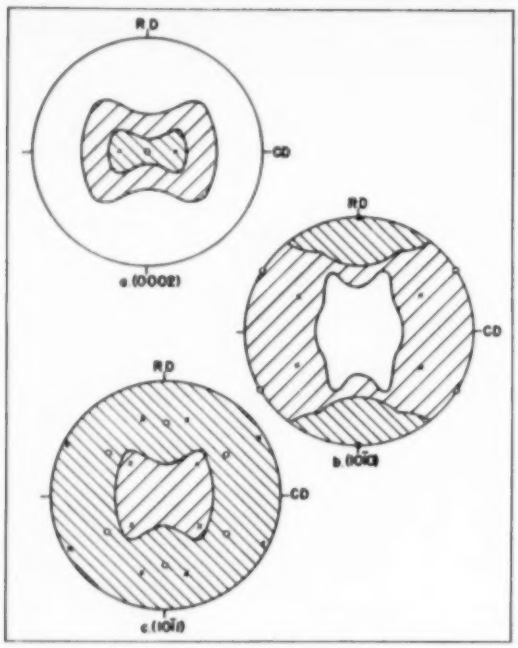


Fig. 6—Iodide titanium hot rolled at 1050°F. o = (0002) [10$\bar{1}$0]. x = (0002) [10$\bar{1}$0] tilted 27°.

The orientation (0002) [10$\bar{1}$0] is marked by "o" and this orientation tilted 27°, as in the cold rolled textures, is marked by "x." It can be seen that all regions are satisfactorily explained on the basis of (0002) [10$\bar{1}$0]. It would be possible to describe the hot rolled sheet texture as a great amount of spread about the cold rolled texture. However, it is felt, on the basis of Figs. 6b and c, that the spreads of orientations are better described as deviations from (0002) [10$\bar{1}$0].

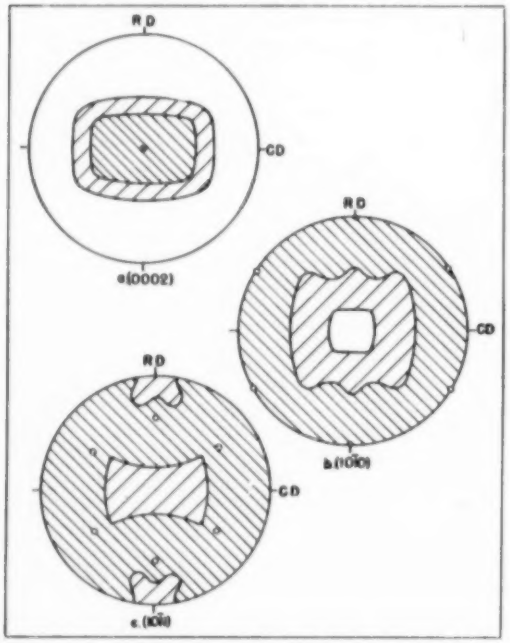


Fig. 7—Iodide titanium hot rolled at 1450°F. o = (0002) [10$\bar{1}$0].

Hot Rolled Texture at 1450°F: As Fig. 7 shows, the randomness of the texture in the specimen rolled at 1450°F is greater than in that rolled at 1050°F. However, the texture still retains the average position of the (0002) plane parallel to the rolling plane. There is a spread of the (0002) plane ±30° in the rolling direction and ±40° in the cross direction from this ideal orientation. In this texture, no one direction appears to be preferentially aligning parallel to the rolling direction.

Effects of the $\alpha \rightarrow \beta$ Transformation on Textures: It can be seen in Fig. 8 that a sharp texture is present after the double transformation $\alpha \rightarrow \beta \rightarrow \alpha$. This texture can be described by the 27° tilted basal plane with the [11$\bar{2}$0] direction parallel to the rolling direction. This is the same orientation reported for iodide titanium recrystallized at 1500°F.

One specimen of cold rolled titanium sheet was cycled five times between the α and β regions, and X-ray shots were made to determine if any change

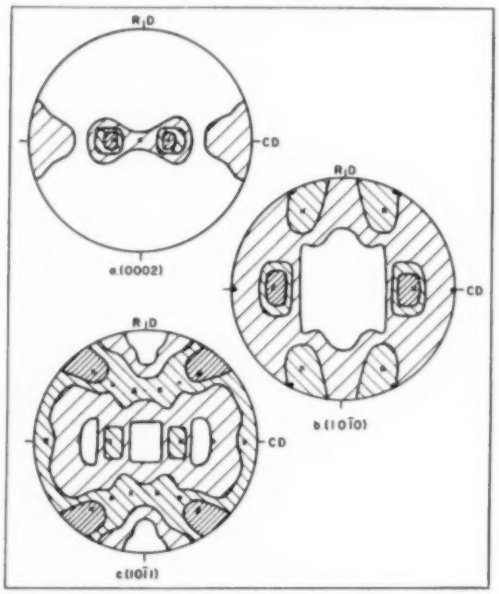


Fig. 8—Texture in iodide titanium cold rolled 97 pct, heated above the $\alpha \rightarrow \beta$ transformation temperature and slowly cooled. o = (0002) [11$\bar{2}$0]. x = (0002) rotated 27° about rolling direction, [11$\bar{2}$0] parallel to rolling direction.

in the texture resulted. The only difference noted was a slight increase in the spread of the basal planes in all directions. Otherwise, the sharp texture of Fig. 8 was retained.

Summary

1—The wire texture for iodide titanium has the [10$\bar{1}$0] direction parallel to the wire axis in the cold rolled state. Recrystallized wires have an average orientation containing the [11$\bar{2}$0] direction parallel to the wire axis, but the actual orientation has the normal to the (21$\bar{3}$0) plane parallel to the axis.

2—The cold rolled sheet texture is described as having the (0001) planes tilted approximately 27° in the transverse direction to the rolling plane and the [10$\bar{1}$0] directions parallel to the rolling direction.

3—Sheets annealed in the vicinity of 1000°F retain the cold rolled textures. Those annealed at

1300°F show a component explained by the cold rolled texture, and one explained by this texture rotated 30° about the normal to the basal plane, i.e., [11$\bar{2}$0] parallel to rolling direction. Annealing at 1500°F produces only the texture containing [11$\bar{2}$0] parallel to the rolling direction and the (0002) tilted 27° to the rolling plane.

4—Hot rolling at 1050°F develops a (0002) [10$\bar{1}$0] texture with spread more pronounced in the transverse than the rolling direction. Rolling at 1450°F aligns the (0002) plane parallel to the rolling plane, but does not show a preferred direction parallel to the rolling direction.

5—The texture present after the double transformation $\alpha \rightarrow \beta \rightarrow \alpha$ is the same as the high temperature recrystallization texture.

Acknowledgment

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Technical Note

Torsion Texture of 70-30 Brass and Armco Iron

by W. A. Backofen and B. B. Hundy

THE pole figure interpretation in a recent paper[1] on the torsion texture of copper was questioned in discussion,[2] and a simpler alternative interpretation was proposed. In the hope of reaching a positive decision in favor of one or the other, further studies were undertaken with 70-30 brass and Armco iron. This additional work has not answered all the questions about torsion textures, but the new pole figures do offer support for the original interpretation and show that the texture is more complicated than the alternative suggests.

From the previous paper,[1] the appropriate method of describing the texture in a twisted bar is known to be a pole figure plotted on a plane tangent to the surface of the bar. The axes of the pole figure are chosen to coincide with directions in the tangent plane which are parallel and at right angles to the bar axis (the longitudinal direction, L.D., and the transverse direction, T.D.), for these directions define symmetry axes of the texture. Therefore any ideal orientation that might be selected to represent the texture gives the crystallographic plane parallel to the tangent plane and the crystallographic direction parallel to the axis of the torsion specimen.

A unique texture was assumed in the original interpretation, and the four ideal orientations, (112) [11$\bar{1}$], (11$\bar{1}$) [112], (110) [001], and (112) [1$\bar{3}$1], were used in its description. Of the four orientations, the first three provide possible slip directions along the direction labeled T.D. in the pole figure, which is one of maximum shear stress in a specimen being twisted. The alternative, however, simply considered the texture to be made up of two components, one a [111] alignment along the specimen axis with a random orientation about the axis as in the wire texture, and the other a superimposed orientation (110) [001]. Experimental evidence for this simpler interpretation was derived from two observations: 1—twisting did not alter the orientation of a single-crystal torsion specimen of α brass in which an octahedral plane was perpendicular (and a [111] direction parallel) to the axis, and 2—deformation bands with approximately the (110) [001] orientation were seen to develop in the crystal during the test.

The new pole figures were obtained with a brass and an iron torsion specimen of $\frac{3}{4}$ and 1 in. gage length diameter, respectively. After annealing, polishing, and twisting, seven rods, about 0.030 in. in diameter and with different orientations, were prepared from material near the surface of each specimen; these were required for the method of constructing pole figures by using a Geiger counter as developed by Norton.[3] Since the shear strain gradient in plastic torsion is linear, it was readily computed that the material supplying the brass rods had been subjected to a shear strain of about 3.2, and that supplying the iron rods had received a shear strain of about 2. All details concerning specimen design, testing procedure, rod preparation, and use of the Norton technique can be found in ref. 1. Only this procedure was followed in working out the (111) and (100) pole figures for brass, Figs. 1 and 2. However, X-ray photograms were also involved in the construction of the (110) pole figure for Armco iron, Fig. 3. The photograms were made according to the conventional transmission diffraction technique using a small sheet taken parallel to the specimen surface and etched to a thickness of about 0.0025 in. They were especially important in locating the boundaries

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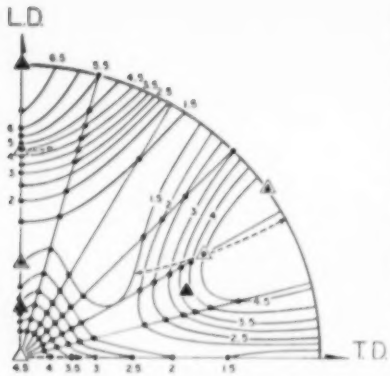

Fig. 1—(111) pole figure of 70-30 brass twisted to a shear strain of 3.2. Ideal orientations indicated as follows: Solid triangle, (112) [11$\bar{1}$]. Open triangle, (11$\bar{1}$) [112]. Double triangle, (110) [001].

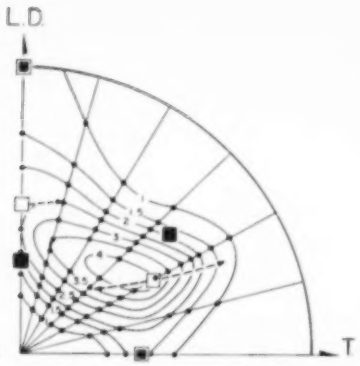

Fig. 2—(100) pole figure of 70-30 brass twisted to a shear strain of 3.2. Ideal orientations indicated as follows: Solid square, (112) [11$\bar{1}$]. Open square, (11$\bar{1}$) [112]. Double square, (110) [001].

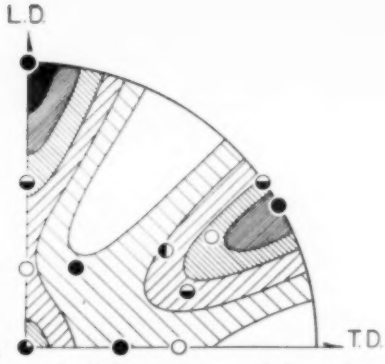

Fig. 3—(110) pole figure of Armco iron twisted to a shear strain of 2. Ideal orientations indicated as follows: Solid circle (111) [1$\bar{1}$0]. Open circle (112) [1$\bar{1}$0]. Vertical half-solid circle, (110) [1$\bar{1}$0]. Horizontal half-solid circle, (110) [1$\bar{1}$2].

of the areas of lowest pole density. Because of the combination of methods, the pole distribution in Fig. 3 has been represented with a precision intermediate between that warranted by the counting data and photograms.

Three ideal orientations, (11$\bar{1}$) [112], (112) [11$\bar{1}$], and (110) [001], have been selected for the simplest approximation of the torsion texture of brass. The first two help to account for the high concentrations of [111] poles in the polar region, the central region, and, to an important extent, the peripheral region about 60° to 75° from the longitudinal direction; they also position poles reasonably well in the (100) pole figure. The (110) [001] is likewise required by both pole figures, although it is not a particularly strong component of the texture. All three provide a [110] slip direction along the transverse direction of maximum shear stress. There is, of course, much spread about these orientations. A possible rotation of approximately 22° around the [112] direction of the (11$\bar{1}$) [112] orientation is indicated with dashed lines in Figs. 1 and 2. Rotations about the [11$\bar{1}$] and [001] directions of the other two orientations are much more limited, however. Rotation about the transverse direction is also possible. The (112) [11$\bar{1}$] can be rotated into the (11$\bar{1}$) [112] and then the (110) [001], although orientations between the last two are not prominent. Further rotation is possible with the (11$\bar{1}$) [112] and (112) [11$\bar{1}$] around the poles of the (11$\bar{1}$) and (112) planes. There is no need for the fourth orientation, (112) [13$\bar{1}$], used in the description of this texture in copper. Considering only the (111) pole figure, the texture might still be interpreted as duplex, consisting of a [111] wire-like component plus the orientation (110) [001]; but this possibility is eliminated by the nature of the (100) pole figure.

The torsion texture of iron appears to consist of a strong alignment of [110] directions along the specimen axis, which is limited in its range of orientation around the axis, with (110) [1$\bar{1}$2] superimposed. The spread of the [110] alignment around the longitudinal direction might be taken as the angle between the [110] pole of a (110) [1$\bar{1}$0] orientation and [112] pole of a (112) [1$\bar{1}$0] orientation, or approximately 54°. It seems more reasonable, however, to rule out, or at least not emphasize the (110) [1$\bar{1}$0] limit and replace it with something closer to (111) [1$\bar{1}$0]. Then the spread would be nearer 20°, roughly the angle between [111] and [112] poles. By slighting the (110) [1$\bar{1}$0] orientation, fewer poles from the alignment along the specimen axis appear in the central area of high pole density, which is then accounted for by the (110) [1$\bar{1}$2] orientation. The latter orientation also places poles in the peripheral area of high intensity, and their contribution on top of that from the [110] alignment explains fairly well the intensity variation in that area. Rotations about the transverse direction and an axis perpendicular to the plane of the figure are also possible here. For example, a rotation might be made about a [110] direction perpendicular to the figure to convert (110) [1$\bar{1}$2] into (110) [1$\bar{1}$3]. Both the (112) [1$\bar{1}$0] and (110) [1$\bar{1}$2] place a [111] slip direction along the transverse direction of maximum shear stress. And again there appears to be more than a superficial difference between the torsion texture and wire texture.

A few photograms were also taken using transmission specimens prepared from two other bars of brass and iron that had been twisted and untwisted by equal amounts. These indicated that the texture established by twisting is not altered if the direction of twisting is reversed, which is the conclusion reached in the work with copper from comparison of complete pole figures.

To summarize, according to the new pole figures, the torsion texture is not the texture found in cold-drawn wire; it is a unique texture that can be described approximately by a combination of ideal orientations, but one that requires still more study before generalizations about its development, description, and correlation with flow can be made.

Acknowledgment

The authors are indebted to the Office of Naval Research for support of this work and to Walter R. Hibbard, Jr. for reviewing the results.

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Forging of Arc-Melted Chromium

by H. L. Gilbert, H. A. Johansen, and R. G. Nelson

High purity electrolytic chromium plate has been hydrogen-reduced and arc-melted under inert atmosphere to give sound ingots. These ingots may be hot forged to break the as-cast structure and then worked at 500°C to finished form. An intermediate and final high temperature anneal gives best ductility and grain refinement.

THE increasing interest in refractory metals for use in high temperature gas turbines, jets, and rockets has led to great efforts in exploiting the fifth and sixth groups of transition elements. Much of the interest has centered upon production of metals that can be worked and fabricated by normal means to produce various engine parts or components having the high temperature characteristics of oxidation resistance and high creep strength required for such uses. If these pure elements do not exhibit all of the desired characteristics, it may be possible to obtain a high purity alloy that will.

Since, in all cases, development of these transition elements has depended to a large extent upon removal of oxygen and nitrogen as a means for producing ductile metal, the hypothesis has been accepted that oxygen is the normal embrittling material in a high grade electrolytic chromium metal. Various methods have been employed in an effort to remove this oxygen by direct or indirect attack.[1]

Many workers have endeavored to produce pure chromium through reduction of the oxide by carbon or active metals, while others have recommended fusion electrolysis of chromium salts or reduction of these salts by active metals, such as sodium or calcium. Hydrogen reduction of the oxide or chloride and thermal dissociation of the iodide have been reported to give high purity metal.

Experimental Work

The U. S. Bureau of Mines, Northwest Electrodevelopment Laboratory, has systematically reexamined the more promising methods and developed two which produce metal having ductility at temperatures lower than heretofore described.

It has been found[2] that oxide-free chromium chlorides in a NaCl-KCl carrier salt may be magnesium-reduced and the byproduct salts distilled off in vacuo to give a high purity chromium sponge. Fusion analysis conducted under the supervision of W. W. Horton of Knolls Atomic Power Laboratory showed the oxygen and nitrogen both to be below 10 parts per million on a selected sample. A small amount of carbon is carried over in the final metal from the chlorination and reduction steps which are carried out in graphite crucibles. This trace of carbon is not detrimental to hot ductility.

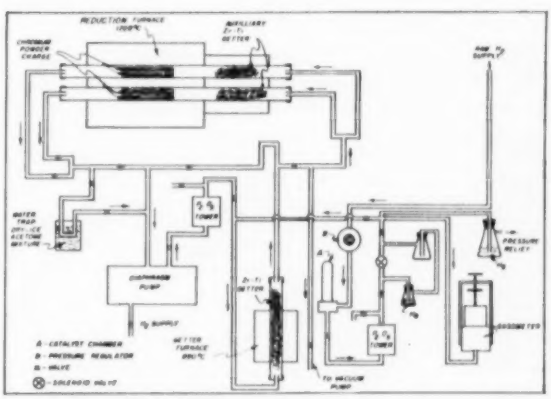

Fig. 1—Hydrogen reduction equipment for chromium powder.

A more practical method for producing ductile metal is through hydrogen reduction of high purity electrolytic chromium. Normal electrolytic chromium will, if "developed" for a few minutes at 1100°C in high vacuum, be found by the method of Adcock[3] to contain about 1 pct chromium sesquioxide (Cr_2O_3). This small amount of oxygen may be effectively reduced by hydrogen.[3, 4]

The method employed was to crush electrolytic chromium plate in a gyratory crusher to pass 60-mesh; finer crushing appears to offer no advantage. A nitric acid leach removed the iron picked up during crushing. The leached powder was rinsed until neutral with distilled water and vacuum-dried.

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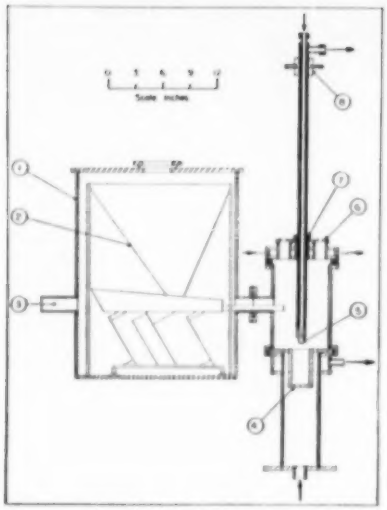

Fig. 2—Description of arc melting furnace.

1—Vacuum tight feed tank
2—Vibrating feeder
3—Vacuum connection
4—Melting cup
5—Thoriated tungsten electrode
6—Eyepiece
7—Packing gland for electrode movement
8—Electrical connection, dc, cathode.

Charges of chromium powder were loaded in molybdenum boats into the 2-in. diam porcelain tubes of the reduction furnace shown in Fig. 1. The system was then evacuated and checked carefully for leaks. Cylinder hydrogen was admitted through a palladium catalyst tower to convert contained oxygen to water which was removed in the phosphorus pentoxide tower. Hydrogen was added until the gasometer rose, which insured a pressure above atmospheric in the system.

Rapid circulation (25 liters per min at standard temperature and pressure) was accomplished by means of a small commercial diaphragm pump sealed in a steel box under an atmosphere of hydrogen from the furnace system so that pump leakage was no problem. The getter furnace was heated to effect initial purification of the circulating hydrogen gas. A Zr-Ti alloy (50-50 mol pct) getter that has a very high affinity for any contaminating gas present was employed. The cold trap was then filled with dry-ice-acetone mixture and the reduction furnace turned on. Most of the water formed by reduction was removed from the system by this cold trap, so the metallic getter and phosphorus pentoxide traps had a long service life.

The temperature of the reduction furnace was maintained at a selected point between 1100° and 1200°C. The main getter furnace was operated at 890°C, and small auxiliary furnaces at the inlet end of the reduction tubes were maintained at a like temperature. Considerable hydrogen was absorbed by the getters when they were first exposed, but no further absorption or elimination was encountered under the conditions of operation. After 16 hr at reduction temperature, the furnace was cooled and the metal removed from the molybdenum boats. As some sintering of the chromium powder took place during reduction, it may be sized upon removal. Lumps ¼ to ⅛ in. in size were found best for arc-melting purposes.

Melting was accomplished in an inert-atmosphere arc furnace shown in Fig. 2. A thoriated tungsten tip ½ in. in diam and 1 in. long screwed into a water-cooled copper electrode holder was used. The melting crucible was of pure copper approximately 2 in. ID and 4 in. long. The chromium lumps were loaded into the feed box and the furnace evacuated and backfilled with 80 pct helium-20 pct argon from a gettered supply tank. The chromium was fed to the melting cup by a built-in vibrating feeder. An arc of 40 v and 800 amp (dc) will melt 750 g of chromium in 20 min. The ingots as removed from the furnace had a fairly good outer surface, but removal of ⅛ in. depth on the lathe assured that no cold-shut difficulties would be encountered. Three sizes of ingots are shown in Fig. 3.

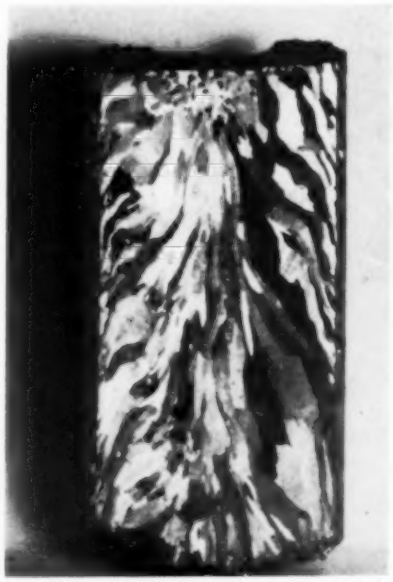

Fig. 4—Half section of chromium ingot, full scale. Etchant, aqua regia.

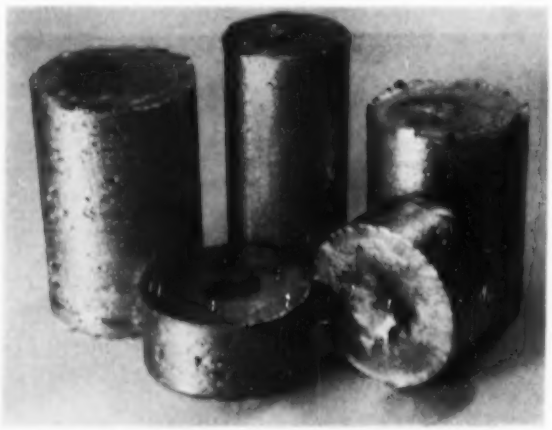

Fig. 3—Arc melted chromium ingots, 1⅛ to 2 in. diam.

Representative analysis of the metal is as follows: Fe, 0.001 pct; Si, 0.05; O, 0.003; N, 0.002; and H, 0.005. In regard to all other metallic impurities, the metal is as pure as the best spectrographic standards available on the market today. Tungsten pick-up during melting was nonuniform and varied from none to

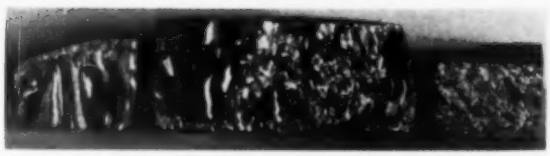

Fig. 5—Chromium ingot. Etchant, aqua regia. X2. Area reduced approximately 75 pct for reproduction. a (left)—As cast. b (center)—Forged 25 pct at 850°C. c (right)—Forged 25 pct, annealed 1 hr at 1200°C in hydrogen.

0.01 pct. Tungsten distribution was very random, and it may be eliminated by use of a consumable electrode of chromium.

Average ingot hardness in the as-cast condition was R_b 55 and in the annealed condition R_b 46.

The ingots were taken from the arc furnace and given a hydrogen anneal for 1 hr at 1200°C, as severe strains are set up during cooling. After annealing and scalping the ingots may be worked open or sheathed. Owing to the small size of the ingots it was preferred to sheath them in iron pipe to pre-

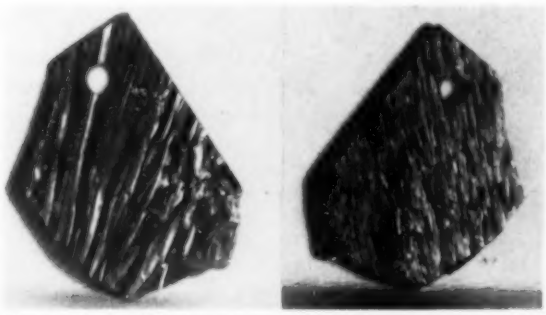

Fig. 6—Sheet rolled from ingot without anneal. Etchant, aqua regia. X1.5. Area reduced approximately 70 pct for reproduction. a (left)—As removed from sheath. b (right)—Polished and etched.

vent chilling of the chromium on contact with the cold forging hammer or rolls. Initial forging was accomplished at 800° to 850°C with moderate reductions until the cast structure was broken up. This may be followed by heavier reductions at 500°C. After the material had been reduced to final dimension, an anneal in hydrogen at 1200°C gave maximum ductility and grain refinement. This material, however, cannot be bent at room temperature without fracture.

It may be seen that this treatment resembles the techniques employed for both arc-melted vanadium[4] and molybdenum,[7] although in some respects the behavior of the metal is more like that of tungsten.

Fig. 4 shows a sectioned arc-cast chromium ingot and Fig. 5 the grain refinement effected by forging 25 pct at 850°C followed by a 1 hr anneal at 1200°C. If the arc-cast ingot is worked to finished sheet without an intermediate recrystallization, a very coarse, fibered structure results as shown in Fig. 6. Reductions in area as high as 93 pct have been effected by rotary swaging. As with forging and rolling, an intermediate recrystallization was beneficial in avoiding a coarse-grained product. A rod swaged at 950°C is shown in Fig. 7.

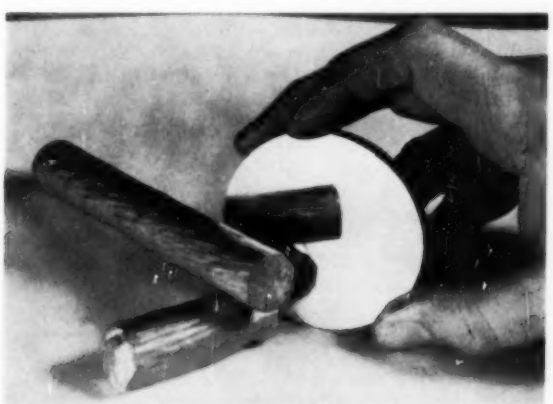

Fig. 7—Swaged rod ¾ in. diam and forged and polished disk of chromium.

It is felt that the status of development of pure chromium metal is encouraging; but, in view of the lack of cold ductility, despite the many efforts made to overcome this condition, the possibility remains that low ductility may be an inherent property of the metal.[8] The excellent work on beryllium[9] has given an extruded metal with fine grain and preferred orientation having an elongation of only 5 to 10 pct in the direction of extrusion. Chromium's sister element, tungsten, seldom displays an elongation better than 6 pct except in fine wire, and there is no reason why chromium may not fall in the same class.

It is believed from the results noted herein that chromium may display the final ductility of tungsten and perhaps even reach the very useful position of molybdenum.

Conclusions

It has been found that a hydrogen-reduced electrolytic chromium powder may be arc-melted to prepare ingots for forging, rolling, or swaging to desired form. Finished forms exhibit ductility at considerably lower temperature than heretofore described. It is possible that in such condition chromium will find wider usage in high temperature applications, either pure or alloyed with other metals of equal purity.

Acknowledgment

Acknowledgment is made to W. W. Stephens, Head of the Rare Metals Branch of the Albany Station, under whose supervision this work has been carried out and to R. A. Beall, Head of the Fusion Section, responsible for production of our ingots. Special acknowledgment is made to W. J. Kroll, formerly consultant to this station, whose enthusiasm and guiding genius permeated this work.

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Flow and Fracture Characteristics of The Aluminum Alloy 24S-T4 as Affected by Strain Thermal History

by S. I. Liu and E. J. Ripling

IT has been shown in a number of recent publications that much information on the mechanical behavior of metals can be gleaned by first deforming test specimens under one set of conditions, and then evaluating the effect of this deformation by means of a subsequent tensile test. A straining procedure of this type has been applied to study the flow behaviors of a number of metals.[1-3] In these studies the prestraining and testing were both tensile strains, while the straining temperature was varied between the prestraining and testing steps. These data indicate that there is a more or less general flow behavior exhibited by all metals which are prestretched at one temperature and then tested in tension at a second temperature. Prestraining a metal at a high temperature produces a lower yield strength during the subsequent low temperature strain than would the same total strain at the lower temperature. On reversing the order of the strains, low temperature prestraining produces a higher yield strength during the subsequent high temperature straining than would a single high temperature strain of the same magnitude.

Unlike the flow behaviors, the fracturing characteristics of metals when prestrained under one set of conditions and tested under a second set of conditions is quite material dependent. The fracture behavior of a number of steels[2,3] as well as of a commercially pure zinc,[4] have been investigated by these two-step tests and was found to depend upon the position of the transition temperature with respect to the prestraining and testing conditions. Since these fracturing phenomena are complicated by the appearance of the transition temperature, it has been difficult to reliably define the basic effect of a two-step straining procedure in fracturing.

Metals crystallizing in the face-centered cubic system do not exhibit transition temperature effects so that the mechanical characteristics obtained on these materials could serve as a base for two-step straining behaviors. Consequently groups of specimens of the aluminum alloy 24S-T4 were prestrained various amounts at room temperature in either tension or compression after which these strained specimens were further strained to failure, or tested in tension, at either room or some lower temperature. Other groups of 24S-T4 specimens were prestretched at various low temperatures after which these were tested in tension at room temperature. These straining techniques then made it possible to compare the behaviors of aluminum with the behaviors previously presented for steels.

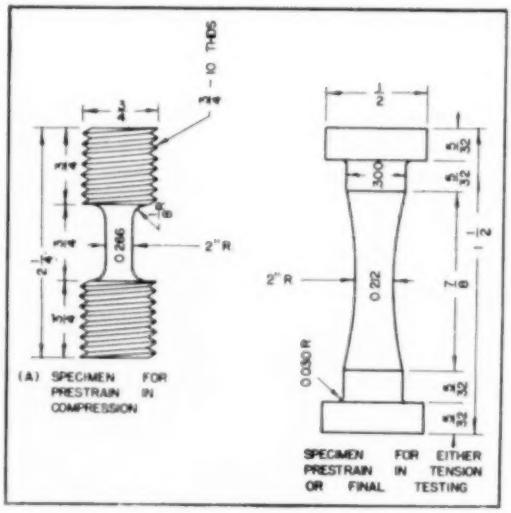

Fig. 1—Test specimens.

Material and Procedure

The commercial aluminum alloy 24S-T4 in the form of ¾ in. rods was used in this investigation. The material was subjected to the following treatments before machining: 1—resolution treating at 920° ± 10°F for 45 min, 2—quenching in water at room temperature, and 3—aging at room temperature for four days.

The re-heat treated material was then machined to either of the specimen shapes shown in Fig. 1. Fig. 1a shows the threaded-end specimen used for prestraining in compression. Fig. 1b shows the specimen used for both prestrain in tension and final testing. The material subjected to precompression in the form of the specimen shown in Fig. 1a was remachined to the specimen shape in Fig. 1b for final testing. Prestraining in tension and final testing were done without any intermediate machining.

All straining was performed on a 10,000 lb Riehle tensile testing machine. Tensile strains were conducted in a specially designed concentric fixture which produced an eccentricity of less than 0.001 in.[5] The change in diameter at the minimum section of

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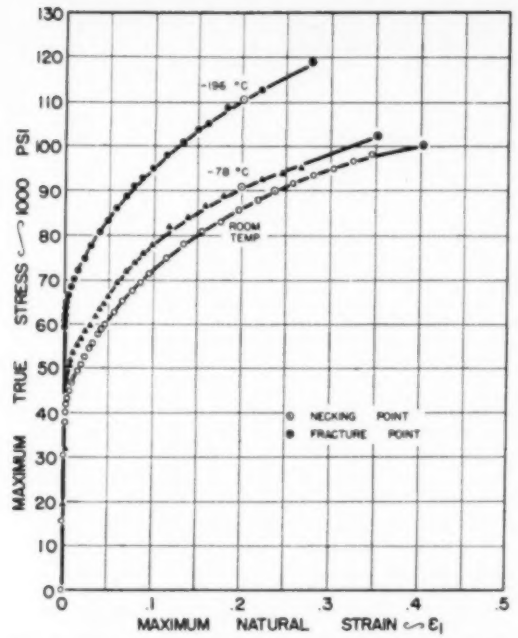


Fig. 2—Stress-strain curves of the aluminum alloy 24S-T4 at different testing temperatures.

the specimen during straining was measured by a radial strain gage reading in ±0.0001 in.[5] A strain rate corresponding to a movement of the test machine head of approximately 0.05 in. per min was used for all strains. The concentric tensile testing equipment was modified for low temperature testing as previously described.

Results and Discussion

The effect of testing temperature on the stress-strain behavior of the aluminum alloy is shown in Fig. 2. Although the difference between the fracture ductility at room temperature and −196°C is not nearly as large as is the case for most steels, the difference is considerably greater than has been reported for a number of other face-centered cubic metals.[6]

Prestraining the aluminum alloy at room temperature and testing at −78° or −196°C produced a lower flow stress curve than would the same total strain at the low temperature. The data obtained by testing at −196°F are shown by the curves in Fig. 3. Again, prestretching at the low temperature produced a higher room temperature flow curve than would an equal strain at room temperature, Fig. 4. These curves are quite like those previously obtained on 2S-O aluminum and copper.[1]

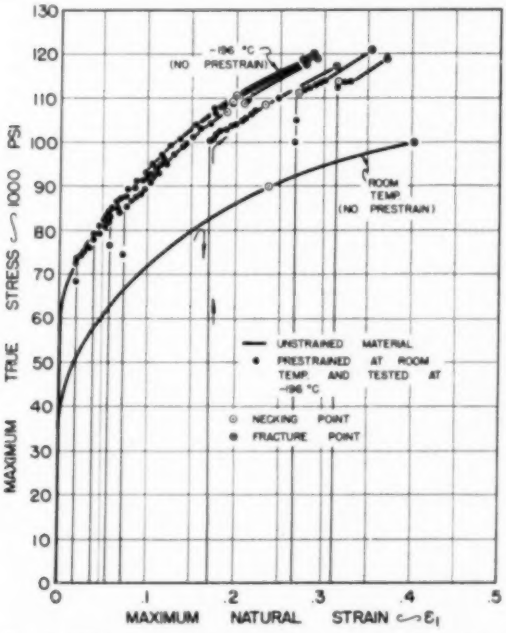

-196 °C (NO PRESTRAIN)
ROOM TEMP (NO PRESTRAIN)
UNSTRAINED MATERIAL
PRESTRAINED AT ROOM TEMP. AND TESTED AT -196 °C
NECKING POINT
FRACTURE POINT
MAXIMUM TRUE STRESS ⟶ 1000 PSI
MAXIMUM NATURAL STRAIN ⟶ ϵ_l

Fig. 3—Effect of prestrain in tension at room temperature on the stress-strain curve in tension of the aluminum alloy 24S-T4 at −196°C.

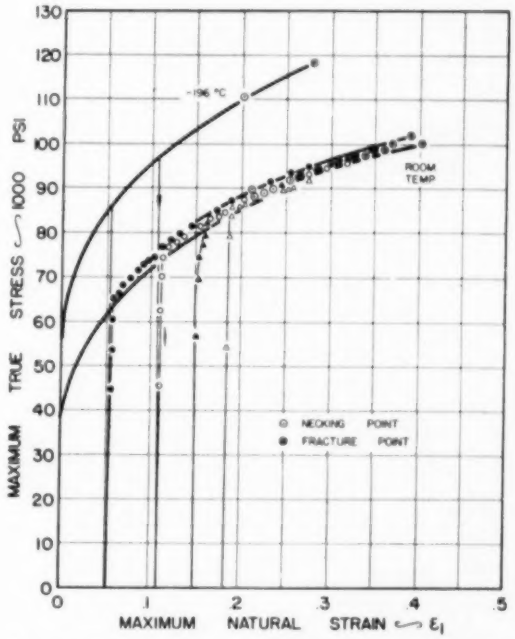


Fig. 4—Effect of prestrain in tension at −196°C on the stress-strain curve in tension of the aluminum alloy 24S-T4 at room temperature.

The fracture stress and fracture ductility possessed by the aluminum alloy at room temperature, −78°, and −196°C after prestraining at room temperature in tension and compression are shown in Fig. 5. Room temperature tensile prestrains apparently have little effect on the low temperature fracture stress, while room temperature compressive prestrains are harmful to the fracture stress only when the magnitude of the precompression is quite large.

When the room temperature prestraining and the low temperature testing were both by tension, the dependence of retained ductility on prestrain can be described by the equation previously presented for steel:

$$\left(\frac{\epsilon_p}{\epsilon_s}\right)^m + \left(\frac{\epsilon_r}{\epsilon_u}\right)^n = 1 \qquad [1]$$

where: ϵ, the maximum natural strain, is defined as the natural logarithm of the ratio of the original cross-sectional area to the final cross-sectional area; ϵ_r, the retained ductility in the second straining op-

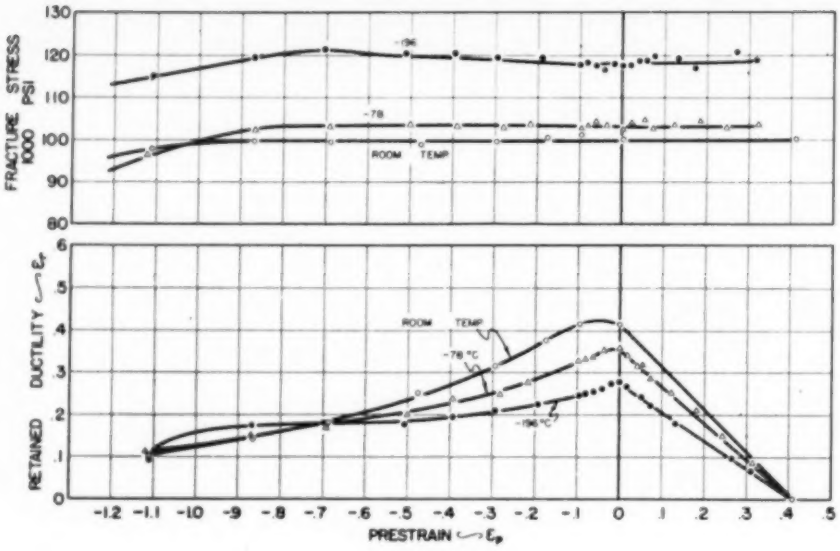
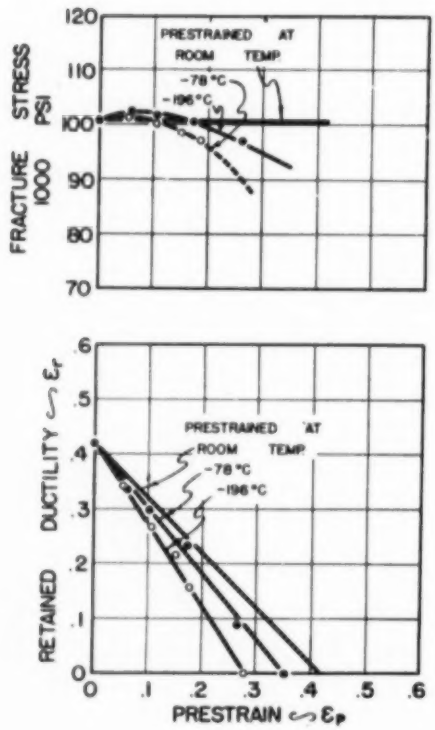


Fig. 5 (right)—Effect of room temperature tensile and compressive prestraining on the low temperature fracture stress and ductility of the aluminum alloy 24S-T4.

Fig. 6 (below)—Effect of low temperature tensile prestraining on the room temperature fracture stress and ductility of the aluminum alloy 24S-T4.



eration; ϵ_1, the fracture ductility at the temperature of first strain; ϵ_2, the fracture ductility at the temperature of second strain; ϵ_p, the prestrain; and m and n, the material constants.

Since the retained ductility-prestrain curves are straight lines in this case, m and n are equal to one, as is the case for most of the materials investigated to date.

Small compressive prestrains at room temperature were again shown to be unharmful to the tensile ductility both at room and low temperatures. An earlier study showed small compressive prestrains to be helpful to room temperature tensile ductility[7] and this effect probably persists even with low temperature straining, although data at sufficiently small strains were not collected.

Reversing the order of strains so that the low temperature stretching preceded the room temperature stretching again produced retained ductility-prestrain curves which were straight lines, intercepting the ordinate at the value of room temperature ductility, and the abscissa at the values of low temperature ductility, see Fig. 6. Hence the behavior is again described by Eq. 1.

Acknowledgments

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Effect of Alloying Elements On The Behavior of Nitrogen in Alpha Iron

by L. J. Dijkstra and R. J. Sladek

IN earlier work the effect of manganese on the general behavior of nitrogen in iron was the subject of a careful examination by Fast.[1] Part of the investigation was made, in collaboration with one of the present authors (L.J.D.), by using the internal friction method. This technique, which has been described elsewhere,[2] showed some significant changes in the properties of Fe-N alloys by the addition of a small amount of manganese namely:

1—Under standard conditions the maximum of the nitrogen peak in pure iron appears around 20°C and the half width is about 27°C. By addition of 0.5 atomic pct Mn a general broadening of the internal friction peak occurs. The half width is increased by about 9°C. Moreover, a shift of the maximum of about 5°C toward a higher temperature takes place, Fig. 2.

2—The precipitation of nitrogen from the supersaturated solution, measured, e.g., at 200°C, was inhibited by the addition of the amount of manganese given in effect 1.

The broadening of the nitrogen peak was confirmed in more recent work[3] on samples which were prepared from Westinghouse Puron iron. The effect of manganese on the rate of precipitation, however, was much less striking than in the earlier work[1] based on Philips high purity iron.

Theory

To explain the first change in properties just referred to, effect 1, the following two assumptions were made:[1] First, every manganese atom in substitutional solid solution creates around it six interstices located between an iron and a manganese atom, as is evident from Fig. 1. These interstices, conveniently called Fe-Mn interstices, have for nitrogen a free energy level which is ΔG^* lower than the normal Fe-Fe interstices between two iron atoms. For such low concentrations of manganese as 0.5 atomic pct, the possibility that two manganese atoms will be nearest neighbors can be disregarded.

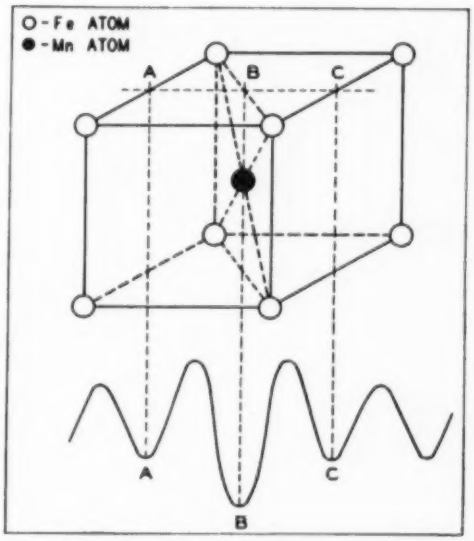

Fig. 1—Assumed modification by a manganese atom of the potential energy of a nitrogen atom in the iron lattice.

Second, it is assumed that in addition to the normal relaxation time τ_{Fe} at least one new relaxation time, $\tau_{Mn\text{-}Fe}$, enters in the phenomena which is associated with the elementary diffusion jumps between a Fe-Mn interstice and an adjacent Fe-Fe interstice. Fig. 1 gives schematically the free energy for a nitrogen atom as a function of its position along the line *ABC*. A tentative analysis of the nitrogen peak in a 0.5 pct Fe-Mn alloy based on these assumptions has been made in Fig. 2. The dotted curve with a maximum at 22°C is caused by jumps between Fe-Fe interstices, the dotted curve with maximum at 32°C by jumps between an Fe-Mn and an Fe-Fe interstice. The two effects added together give the dash-dot internal friction curve for the alloy. In the following these two components will be referred to as the normal peak at 22°C and the abnormal one. (The small secondary peak at

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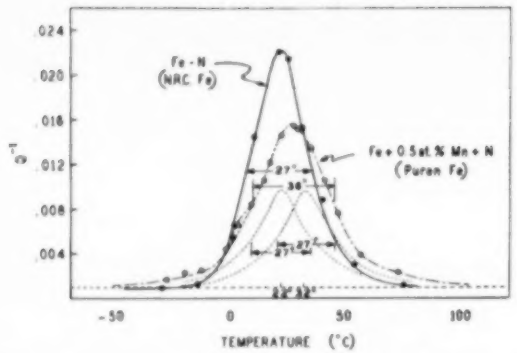

Fig. 2—Influence of manganese upon internal friction due to nitrogen in iron.

3°C is a real effect and increases very much with higher manganese content.)

If by some suitable heat treatment the nitrogen is allowed to precipitate from the solid solution in an alloy, a distinction must be made, in principle, between two different types of equilibria, since the diffusion rates of substitutional and interstitial elements are so widely different. Conditions during precipitation can be such that the alloying element participates in the diffusion, and nitrides of different alloy content from that of the matrix are formed. However, it is also possible that the alloying element does not have an opportunity to diffuse and in that case the nitride inherits its alloy content from the matrix. The terminology introduced by Hultgren will be followed in this paper and the two types of reactions will be called the ortho and the para reactions, respectively.[4] It will also be convenient to distinguish the nitride and nitrogen-solubilities in both cases by the use of the prefixes ortho and para. In this study the para nitride is practically identical with the normal nitride in pure iron and it is clear from the picture, which was given to explain the effect of manganese, that the para solubility $C_{Fe,\,Mn}$ of nitrogen in the Fe-Mn alloy is higher than the normal solubility C_{Fe} of nitrogen in pure iron. The ortho solubility of nitrogen in a 0.5 pct Fe-Mn alloy must still be very small at 600°C. For, after tempering this alloy at 600°C in helium for 1 or 2 hr and quenching to room temperature, the internal friction peak has almost disappeared. A short anneal above 900°C in the γ phase is required to dissolve the ortho precipitate, and a subsequent rapid quench to room temperature reproduces the nitrogen peak to its full, original extent.

By standard methods of statistical mechanics it is found, assuming that the temperature is not so low that saturation of the Fe-Mn interstices with nitrogen occurs, for the para solubility:

$$C_{Fe,\,Mn} = C_{Fe}\,(1 + 2c_{Mn}e^{\Delta G^*/RT}) \qquad [1]$$

c_{Mn} is the atomic concentration of manganese and is assumed to be small. The factor 2 arises from the fact that per manganese atom there are six Fe-Mn interstices and per iron atom only three Fe-Fe interstices. ΔG^* is the increase in free energy on transferring a mol of nitrogen atoms from Fe-Mn sites to Fe-Fe sites (p and T constant) exclusive of the configurational part of the entropy change. The normal solubility $C_{Fe} = 3e^{-\Delta G'/RT}$ where $\Delta G'$ is the free energy of solution per mol of nitride in iron exclusive again of the change in configurational entropy.[5] The fraction of the nitrogen atoms at the Fe-Mn interstices is $2c_{Mn}e^{\Delta G^*/RT}$.

Accepting the analysis of Fig. 2, it follows that at $T = 300$°K, $2c_{Mn}e^{\Delta G^*/RT} \cong 1$ or $\Delta G^* \cong 2800$ cal per mol.

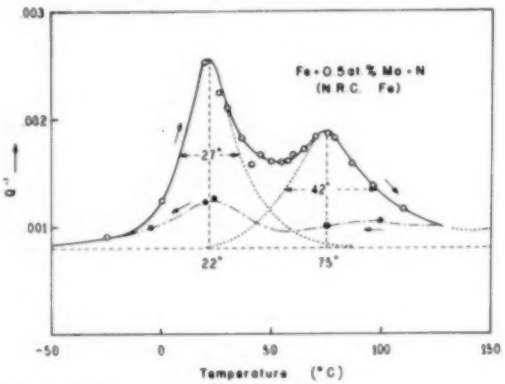

Fig. 4—Influence of molybdenum upon internal friction due to nitrogen in iron.

To explain effect 2, a third assumption appears to be necessary. In general, the rate of para precipitation is governed by three factors, namely: 1—degree of supersaturation with nitrogen; 2—diffusion rate of nitrogen; and 3—number of available potential nuclei.

Using the calculated value of 2800 cal per mol for ΔG^* and substituting this value in Eq. 1, at 200°C the solubility is increased by only 20 pct due to the addition of 0.5 pct Mn. At this temperature, there is only a slight preference for nitrogen atoms to occupy Fe-Mn interstices and therefore the diffusion rate is decreased only by a small amount. The most probable conclusion is that the large influence of manganese on the precipitation of nitrogen in this Philips iron must be a reduction, somehow, of the number of potential nucleation sites.

After cold work (e.g., 50 pct reduction in area) of the Fe-Mn alloy the internal friction peak drops rapidly by aging at room temperature. In other words, dislocations offer a much lower energy level for nitrogen atoms than the Fe-Mn interstices. This is not surprising since it is known[6] that in pure iron the nitrogen concentration in normal solid solution in equilibrium with the nitrogen phase in the dislocations is very small.

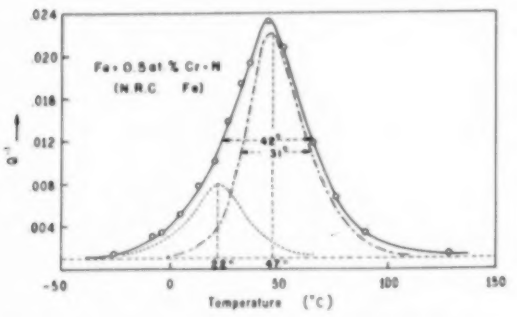

Fig. 3—Influence of chromium upon internal friction due to nitrogen in iron.

Recent Work on Other Alloys

Recently this investigation has been extended to the alloying elements chromium, molybdenum, and

vanadium. All the alloys contained 0.5 atomic pct of the alloying element. In preparation of these alloys National Research Corp. iron was used with an oxygen content of about 0.003 pct. Furthermore, the Fe-Mn alloy was reinvestigated, this time using Westinghouse Puron iron, which has a rather high O_2 content of about 0.1 pct. In all cases the nitrogen was introduced by nitriding at 600°C for 6 min. The alloys were homogenized, unless otherwise indicated, by an anneal at 1000°C in helium for some minutes and then quenched from this temperature. After this treatment the Fe-V-N and Fe-Mo-N alloys turned out to be very unstable and even at room temperature precipitation was very rapid.

In Figs. 2 to 5 the internal friction Q^{-1} is plotted as a function of the temperature for the various alloys. The Fe-Cr alloy was not homogenized at 1000°C. The dotted curves are single relaxation peaks and are the proposed components of the total internal friction curve. In a first approximation analysis into two single relaxation peaks is indeed possible. The low temperature peak is the normal nitrogen peak at 22°C. The abnormal peak introduced by the addition of the alloying elements is

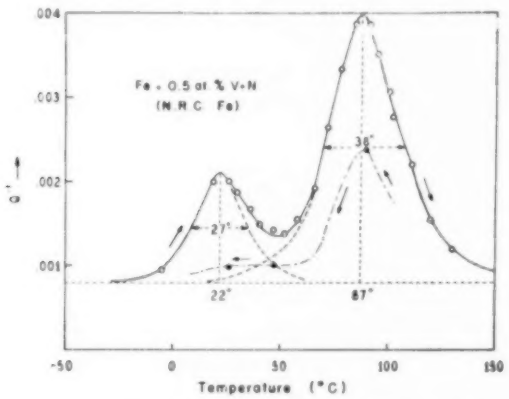

Fig. 5—Influence of vanadium upon internal friction due to nitrogen in iron.

always at a higher temperature. Apart from the theoretical picture given, the analysis in two single relaxation peaks is more or less justified by the curves of the Fe-Mo and Fe-V alloys in which indeed two pronounced peaks occur. The nitrogen peak in the Fe-Mn alloy in Fig. 2 is the exact reproduction of the corresponding peak observed earlier in the Philips iron.[1]

Internal friction measurements have two disadvantages. In the first place, since the temperature is changed during the run, the ratio between the number of nitrogen atoms at Fe-Fe and Fe-alloying metal interstices is not constant and the simple analysis in two single relaxation peaks is not quite correct. In the second place, the rapid precipitation is sometimes very disturbing.

The runs of Q^{-1} vs temperature were made in the direction of increasing temperature. On cooling down from the highest measuring temperature, the Q^{-1} was much lower, especially in the Fe-Mo and Fe-V cases; apparently above 100°C para precipitation takes place during the time of measurement. Therefore elastic after-effect measurements were carried out on the Fe-Mo and Fe-V alloys at 0° and 10°C. The method and apparatus which were used

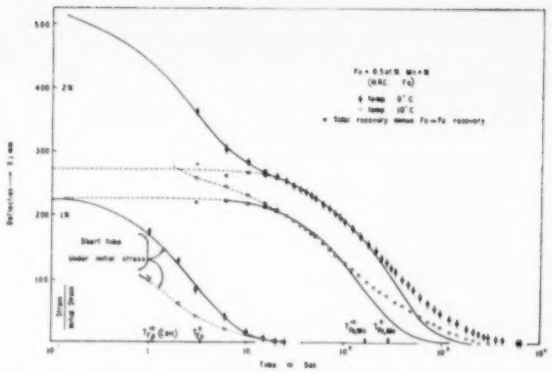

Fig. 6—Influence of molybdenum upon elastic recovery due to nitrogen in iron.

are described in a paper by Wert.[7] The results are shown in Figs. 6 and 7. The ratio of strain over initial strain (that is, the strain while the external constant stress is applied) is plotted against the logarithm of the time which has passed since the external constant stress has been removed and during which strain recovery has occurred.

In general, the results obtained by the internal friction method are at least qualitatively confirmed insofar as mainly two relaxation times enter in the phenomena. The drawn curves correspond to single relaxation times. The normal effect resulting from jumps of nitrogen atoms between Fe-Fe interstices can be measured separately by keeping the specimen under the constant initial stress only for a time comparable to τ_{Fe}, which is therefore much shorter than $\tau_{Mo,\,Fe}$. These separately measured curves are shown in the lower left corner of Figs. 6 and 7. They show a discrepancy in that τ_{Fe} at 10°C is not smaller than 0°C, a discrepancy which shows up for both alloys. The accuracy of measurement is not very great for these short times. For the Fe-V alloy the abnormal effect is larger at 10°C than at 0°C. The opposite would be expected. A possible explanation is that at 0°C a state of thermodynamical equilibrium was not quite realized. In order to establish this equilibrium during cooling, the nitrogen atoms must be allowed to escape from the alloying metal atoms. They also have to diffuse over a range of the order of the average distance between the atoms of the alloying element. Because of the many uncertainties involved, more than a qualitative significance should not be attached to these results and

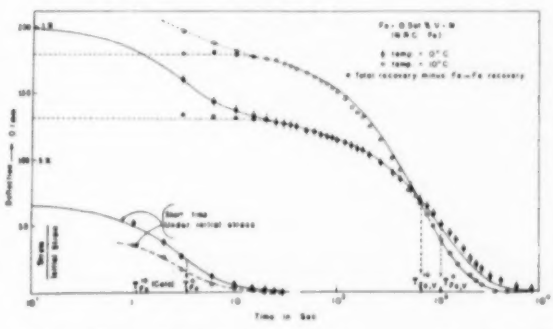

Fig. 7—Influence of vanadium upon elastic recovery due to nitrogen in iron.

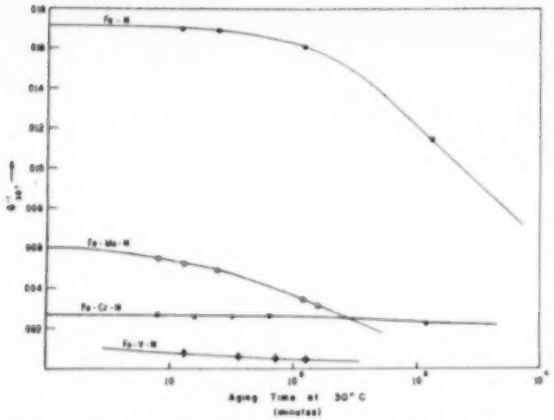

Fig. 8—Effect of alloying elements upon precipitation of nitrogen in N.R.C. iron. Iron quenched from 1000°C before aging.

therefore a further analysis was not made of the internal friction peaks and after-effect curves.

Precipitation

In view of previous results on Philips iron, it was surprising that in this investigation it was found that the rate of precipitation of nitrogen in Puron iron was not affected at all by the addition of 0.5 atomic pct Mn. Nitrogen precipitates for equal concentrations at about the same rate in Puron iron, National Research Corp. iron, Philips iron, and the Puron Fe-Mn alloy. Apparently the high O_2 content of the latter alloy neutralizes the effect of 0.5 pct Mn. The mechanism for this effect is not clear.

Fig. 8 shows the rate of para precipitation of nitrogen at 30°C in various alloys which were all prepared from National Research Corp. iron. The specimens were homogenized at 1000°C in helium for at least 2 min and then quenched from this temperature. Since it was believed that the height of the normal component is the real measure for the degree of supersaturation, the value of Q^{-1}_m as plotted is the height of this component at 30°C. Though no nitrogen analysis has been made, it seems that the Fe-Cr alloys behave abnormally and show a very high rate of precipitation.

Not much information is available on the ortho precipitation reaction. The following was found for the Fe-V alloy only: Tempering at 600°C for 3 min and quenching to room temperature showed a drop in Q^{-1} of about 50 pct; tempering for 45 min, a drop of about 100 pct; tempering at 700°C for 3 min was sufficient to cause a drop of 100 pct in Q^{-1}. This ortho precipitate is probably submicroscopic at 600°C, for vanadium diffuses very slowly at this temperature. The ortho nitride is stable at 700°, 800°, and 900°C, but starts dissolving at 1000°C in the γ phase. The high rate of precipitation in the Fe-Mo and Fe-V alloys at room temperature might somehow be connected with an insufficient quenching rate through the region 900° to 600°C, where a very finely dispersed ortho precipitate is formed.

Discussion

Apparently the nitrogen atom in solid solution in the body-centered cubic iron lattice is not indifferent to the kind of surrounding metal atoms. If a nitrogen atom is by some means transferred from a monoatomic gaseous state into an interstice in the body-centered cubic lattice, the lattice in the immediate neighborhood of the interstice is strained. It would be expected that the strain energy is larger if the interstice is adjacent to a substitutional foreign atom which is larger than an iron atom, e.g., molybdenum and vanadium. According to Pauling,[9] the atomic diameters of iron, manganese, chromium, vanadium, and molybdenum for a coordination number 8 are 2.46, 2.48, 2.52, 2.62, and 2.72A, respectively. That the nitrogen atoms tend, nevertheless, to cluster around the vanadium and molybdenum atoms could then mean only that a second energy term enters which probably is connected with a redistribution of electrons between nitrogen and metal atoms. It is well known that nitrogen is rather strongly electronegative.

Smithells[10] gives for the heat of formation at 0°K the following values:

$$4Fe\ (\alpha) + \tfrac{1}{2}N_2 \rightarrow Fe_4N + 3{,}860\ cal$$
$$3/2Mn + \tfrac{1}{2}N_2 \rightarrow \tfrac{1}{2}\ Mn_3N_2 + 16{,}730\ cal$$
$$Cr + \tfrac{1}{2}N_2 \rightarrow CrN + 30{,}400\ cal$$
$$Mo + \tfrac{1}{2}N_2 \rightarrow MoN + 16{,}600\ cal$$
$$V + \tfrac{1}{2}N_2 \rightarrow VN + 41{,}650\ cal$$

The plus sign on the right means that the reaction is exothermal. It must be admitted that these heats of formation are not purely representative for the nitrogen-metal affinity, since the metal lattice is loosened up in the compound formation to a certain extent. This can explain the rather low value for molybdenum, which has a heat of sublimation much higher than the other metals in the table. The heats of sublimation of the latter do not differ very much. The general tendency is that, in going to lower atomic numbers in a transition series, the compound is more stable. This can be understood qualitatively from the rule that the ionization potential for the outer electrons in general decreases with decreasing atomic number in a transition series.

A comparison between heat of compound formation and relative height of the abnormal peak of the various alloys is made difficult by the rapid precipitation during the measurments. Also, strictly speaking, the maxima ought to be compared for the same temperature. Nevertheless, these data seem to indicate a definite correlation. It seems reasonable, too, that the abnormal peak in the internal friction will always appear at the high temperature side of the normal peak, since the energy barrier which must be overcome for a nitrogen atom to break away from the foreign atom will be larger than in a normal interstice. In the case of manganese, chromium, and vanadium, it is apparent that the larger the relative height of the abnormal peak, the further the latter is shifted toward a higher temperature.

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Systems Zirconium-Molybdenum and Zirconium-Wolfram

by R. F. Domagala, D. J. McPherson, and M. Hansen

On the basis of metallographic analysis, incipient melting data, thermal analysis work, and X-ray diffraction, phase relationships in the 0 to 50 atomic pct alloy content were carefully resolved. Phase relationships in the higher alloy content regions were outlined. A single intermediate phase, peritectically formed, of the form ZrX_2 was established in each system. A eutectic and a eutectoid were found in the zirconium-rich region of both systems. Appreciable solubility of molybdenum and wolfram was found in β zirconium, but only a slight solubility in α zirconium.

PREVIOUS work on these systems was limited to X-ray investigations of the intermediate phases[1, 2] and preliminary surveys of the zirconium-rich alloys.[3] The diagrams presented were determined principally by metallographic, thermal analysis, and incipient melting techniques. In accordance with the Atomic Energy Commission contract requirements, phase relationships up to 50 atomic pct molybdenum or wolfram were carefully resolved, while only a limited amount of work was done to outline the 50 to 100 atomic pct alloy region.

A description and analysis of the metals used in the preparation of alloys for this study are included in Table I. A "low-hafnium" zirconium crystal bar, produced by the decomposition of a volatile iodide onto a hot filament, was employed for these studies. Westinghouse "Grade 3" crystal bar was used for all alloys. The zirconium, as received, was coated with corrosion product from a standard autoclave test by which its grade designation is determined. This was removed by a sand-blasting and $HF-HNO_3$ pickling technique developed by the Atomic Energy Commission. The bars were cold rolled to approximately 1/32 in. sheet, pickled for iron removal, sheared to ¼ in. squares, washed with acetone, and stored for furnace charge.

Molybdenum sheet 0.003 in. thick, 0.001 to 0.005 in. wolfram wire, and ½ in. wolfram rod crushed to granular form, from Fansteel Metallurgical Corp., were used as alloying stock.

Table I. Analyses of Metals Used

	Metal		
	Iodide Zirconium*	Molybdenum	Wolfram
Nominal purity, pct	99.8	99.9	99.9
Form	Crystal bar	0.003 in. sheet	½ in swaged rod; 0.001 to 0.005 in. wire
Impurities, Pct			
Al	0.007		
Co		0.003 maximum	
Cr	0.002		
Fe	0.03	0.005 maximum	
Hf	<0.05		
Mg	0.0001		
Ni	0.007		
Ti	0.013		
O	0.01	0.045 maximum	
N	0.0087		
C	<0.01		
R_2O_3			0.02 maximum

* Zirconium analysis supplied by The Argonne National Laboratory, Chicago.

Melting Procedure

A nonconsumable electrode arc furnace identical to that described in detail by Hansen et al.[4] was used. The source of power for melting Zr-Mo alloys was a 400 amp dc welding generator, while a 900 amp dc generator was used for preparing Zr-W alloys. Electrode tips of the same material as the alloy addition were employed in both systems, i.e., molybdenum tips were used for Zr-Mo alloys, and wolfram tips for Zr-W alloys. This precluded any possible electrode contamination during arc melting.

Ingots of 20 to 30 g of Zr-Mo alloys were melted under high purity helium in the cavity of a copper block insert in a spun copper crucible. Homogeneity was insured by remelting all ingots four to six times without opening the furnace.

Due to the large difference between the melting points of wolfram and zirconium, direct melting of the charged metals as above generally yielded ingots containing unmelted wolfram particles. This was overcome, after many variations in technique, by melting a pure wolfram charge at high power levels (700 to 900 amp) and slowly adding zirconium to form a master alloy. Such master alloys were sectioned and remelted several times, then crushed to serve as melting stock for alloys of lower wolfram content. Master alloys containing 26, 50, 52, 70, and

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This paper represents a portion of the work on an Atomic Energy Commission-sponsored project, Contract No. AT(11-1)-149, "Phase Diagrams of Zirconium-Base Alloys."

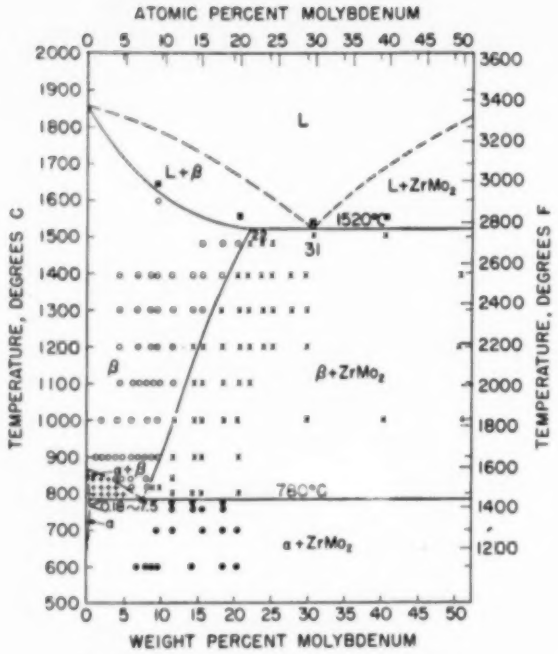

Fig. 1—Partial diagram of the Zr-Mo system.

90 pct W served for the preparation of all the Zr-W alloys. Calculated weights of zirconium were melted with granules of the analyzed master alloy to yield the desired compositions. Alloys containing up to 50 pct W were arc melted in a copper block insert eight times without opening the furnace, while alloys richer in wolfram were melted ten times in a heavy-wall copper crucible. No unmelted wolfram was detected in any of the alloys prepared in this manner.

Annealing Treatments

Previous studies[4, 5] have shown that Vycor and quartz bulbs are suitable for containing specimens of highly reactive alloys of titanium or zirconium during annealing treatments. Vycor was employed for temperatures up to 1100°C, while quartz bulbs were used above this temperature. Evacuated bulbs were employed at temperatures up to 950°C, but for higher temperatures a partial pressure of argon was admitted to the bulb before sealing. This partial pressure was controlled to produce 1 atm of pressure at the temperature of treatment, in order to balance external pressure. Molybdenum sheet 0.003 in. thick was used as a liner in bulbs scheduled for treatments above 1200°C. These liners served to protect the specimens from direct contact with the bulbs and gave physical support to the bulb walls. All annealing treatments except homogenization were carried out in glazed porcelain tubes in electrically heated furnaces. Resistance alloy-wound furnaces were used up to 1250°C, but for higher temperatures a small Globar tube furnace was employed. Temperature control, in general, was ±3°C, but more precise control was achieved for many anneals. Homogenization treatments were carried out in a box-type Globar furnace, with a temperature control of about ±10°C. A control sample of pure zirconium was included in each bulb of isothermally annealed specimens. At the conclusion of an anneal, specimens were quenched by breaking the bulb under water.

Solidus Determinations

A high temperature vacuum-induction furnace was used for the determination of solidus temperatures. A description of the unit, details of operation, and temperature calibration have been given.[4] The technique employed was to heat an alloy of given composition until visual signs of melting were observed, then quench a series of specimens of the same alloy content at approximately 25°C intervals below the temperature of observed melting, and examine them metallographically for evidences of incipient melting.

Thermal analysis was used to check the eutectic temperature in the Zr-Mo system, but was not generally applicable in the present work because of the high temperatures involved. The equipment and techniques employed have been described.[4]

Zr-Mo System

The chemical analyses of alloys prepared for the determination of phase relationships in this system are included in Table II. Table III lists the annealing treatments employed.

Zirconium-rich alloys containing from 0.2 to 28 pct Mo were reduced by cold pressing from 54 to 1 pct, depending upon alloy content, homogenized at 1150°C for 70 hr, and then cold rolled from 75 to 5 pct, prior to isothermal annealing treatments. All other alloys were used in the arc-cast condition.

The phase relationships for the 0 to 50 pct Mo region are presented in Fig. 1, and the entire diagram is shown in Fig. 2. Fig. 1 includes most of the experimental points necessary for the construction of the diagram; some points were omitted for clarity. Preliminary anneals for various times at 700°, 800°, and 900°C indicated that after 250, 150, and 50 hr, respectively, no changes occurred in the relative amounts of phases present in 3, 9, and 16 pct Mo alloys. The results of this program were used to schedule appropriate times for annealing treatments.

It should be noted that a preliminary investigation was undertaken in order to establish the temperature of the allotropic transformation, $\alpha \rightleftarrows \beta$, in "Grade 3" zirconium crystal bar. Specimens from five random bars, after cold pressing, homogenization and cold rolling, were annealed at approximately 850°, 860°, 870°, 880°, and 890°C followed by water quenching. Two phase ($\alpha + \beta$) structures were observed in certain of the specimens for quenching

Table II. Analyses of Zr-Mo Alloys

Alloy Designation	Mo, Wt Pct	Alloy Designation	Mo, Wt Pct
ZM 0.2-727	0.18	ZM 26-317	25.3
ZM 0.4-724	0.35	ZM 28-316	27.9
ZM 0.5-260	0.60	ZM 29-628	28.7
ZM 0.6-725	0.64	ZM 30-323	30.0
ZM 0.8-726	0.77	ZM 31-629	30.7
ZM 1-240	1.2	ZM 32-630	31.3
ZM 2-243	2.2	ZM 33-631	33.1
ZM 3-242	3.1	ZM 34-632	33.8
ZM 4-244	4.2	ZM 35-324	35.4
ZM 5-245	4.7	ZM 40-325	40.6
ZM 6-246	6.2	ZM 45-326	44.8
ZM 7-247	7.1	ZM 50-335	50.9
ZM 8-239	8.3	ZM 55-336	55.7
ZM 9-237	8.9	ZM 60-221	57.8
ZM 10-235	9.6	ZM 62-331	59.0
ZM 12-315	11.9	ZM 64-332	63.5
ZM 14-322	14.5	ZM 66-333	65.4
ZM 16-321	15.7	ZM 68-334	65.4
ZM 18-320	18.5	ZM 70-220	64.4*
ZM 20-319	20.7	ZM 80-219	79.4*
ZM 22-318	22.1	ZM 90-222	89.3*
ZM 24-314	24.0		

* Analyzed for zirconium, molybdenum by difference.

temperatures between 851° and 868°C. Apparently, then, because of impurities, this grade zirconium transforms over a range of temperatures close to the ideal transition point. The value 862°±5°C, reported by Vogel and Tonn,[9] was adopted for construction of the diagrams, but it is certain that this departure from ideality must cause corresponding departures from true binary equilibrium in the very dilute alloys.

The eutectic at 31±1 pct Mo and 1520°±15°C was located by the microscopic examination of several as-cast, incipient melting, and thermal analysis specimens in this region. The temperature was fixed as a result of several heating and cooling curves for an alloy of the eutectic composition, as well as by incipient melting. Fig. 3 shows the eutectic structure in a 30.7 pct Mo alloy, quenched from 1535°C in the incipient melting furnace.

Examination of isothermally annealed specimens showed the solubility of molybdenum in β zirconium to decrease from 22 pct at the eutectic level to 7.5±1 pct at the eutectoid temperature, 780°C. Figs. 4 and 5 represent 20.7 and 22.1 pct Mo alloys, respectively, quenched from 1480°C. A typical retained β structure is evident in the former, while β + $ZrMo_2$ phases are present in the latter structure. Figs. 6 and 7 represent 9.6 and 11.9 pct Mo alloys, respectively, quenched from 900°C. The former is 100 pct β, while the latter contains $ZrMo_2$. The β phase was retained on quenching alloys containing more than 5 pct Mo, but transformed to acicular α (martensite) in alloys of lower molybdenum content.

The eutectoid decomposition: $\beta \rightarrow \alpha + ZrMo_2$ proceeds very slowly in this system. The eutectoid level was determined by examination of hypereutectoid alloys annealed at temperature increments of 15°C. The eutectoid level and the sluggishness of the decomposition are illustrated in Figs. 8 and 9. The former illustrates the retained β + $ZrMo_2$ structure observed after annealing a 14.5 pct Mo alloy at 790°C for 610 hr. The latter figure shows the 15.7 pct Mo alloy quenched from 775°C after 380 hr. In this specimen, a small amount of the β adjacent to proeutectoid $ZrMo_2$ crystals has decomposed. The metastability of β was detrimental to accurate positioning of the eutectoid composition, but the value presented, 7.5 pct, undoubtedly is within ±1 pct of the equilibrium value. Eutectoid decomposition in hypoeutectoid alloys was never observed.

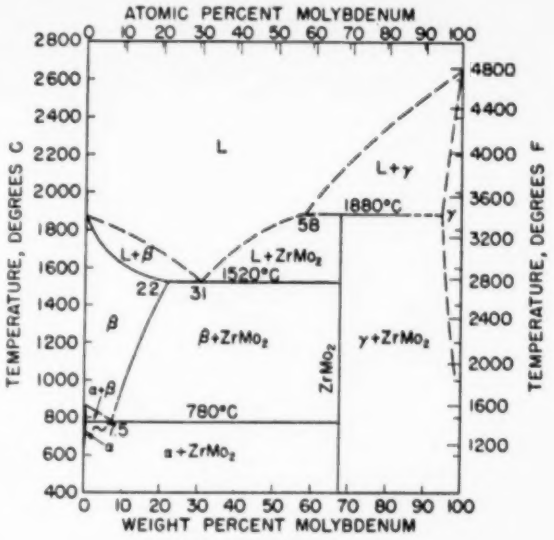

Fig. 2—Zr-Mo system.

The solubility of molybdenum in α zirconium is less than 0.18 pct at all temperatures. Fig. 10 shows a 0.18 pct Mo alloy quenched from 775°C after 240 hr. The α plus metastable β phases are present.

The intermediate phase, $ZrMo_2$, is formed by the peritectic reaction: Melt + molybdenum-rich solid solution → $ZrMo_2$. The peritectic configuration and the composition of the liquid entering into the peritectic reaction are illustrated in Figs. 11 and 12. A 57.8 pct Mo arc-melted alloy is shown in the former figure. Only two phases, $ZrMo_2$ and β, are present. Fig. 12, a 59.0 pct Mo alloy, illustrates the initial appearance of the third phase, molybdenum-rich solid solution, as primary crystals. The composition of the peritectic melt is therefore between 57.8 and 59 pct Mo. Heat treatment in the incipient melting furnace brought the peritectic reaction nearly to completion in a 65.4 pct Mo alloy. $ZrMo_2$ and a small amount of β were present in this structure, supporting the designation of the phase as $ZrMo_2$ (stoichiometrically, 67.78 pct Mo).

An X-ray pattern of −325 mesh powder from a 57.8 pct Mo alloy was made, using filtered, characteristic CuKα radiation. Indexing of the pattern indicated that the intermediate phase has a cubic lattice, C15 ($MgCu_2$ type) with the lattice parameter, a = 7.59Å. This is in agreement with Duwez and

Table III. Schedule of Annealing Treatments for Zr-Mo Alloys

Annealing Temperature, °C	Annealing Time, Hr	Alloys,* Pct Mo
1480	0.2	16-26, 60-64
1400	1	5-30, 50-68
1300	2	5-30
1204	8	5-30, 50-68
1100	20	5-22
1000	40	2-20, 30, 40, 50, 60, 64
900	48	1-20
840	240	0.5, 1-20
830	240	0.2-0.8
815	240	0.5, 1-12
800	240	0.2-0.8
	150	1-20, 40, 50, 62
790	610†	5-18
775	380†	5-18
	240	0.2-0.8
760	380†	5-18
750	240	0.2-0.8
	208	0.5, 1-24
740	610†	5-18
700	335	0.2-0.8
	256	0.5, 1-24, 40, 50, 60, 64
650	400	0.5, 1-10, 14, 18, 22
600	475	0.5, 1-10, 14, 18, 20, 30, 40, 50, 60, 64

* Alloys were pretreated before annealing as described in the text. Control samples of pure zirconium were included in each annealing treatment.

† Specimens held 17 hr at 900°C, slowly cooled to temperature, and held for indicated time, followed by water quench.

Table IV. Melting Range Determinations for the Zr-Mo System

Alloy No.	Mo, Wt Pct	Thermal Arrest, °C	Melting Observed, °C	No Melting, °C
Zr	0		1852	
YZM10-235	9.6		1640	1590
YZM20-319	20.7		1560	
YZM31-629	30.7		1535	1500
XZM31-791	30.8	1520		
YZM40-325	40.6		1550	1505
YZM60-221	57.8		1965	1855
YZM62-331	59.0		1900	1870
YZM66-333	65.4		1900	1870

Fig. 3—30.7 pct Mo alloy quenched from 1535°C. β - $ZrMo_2$ eutectic. Etchant: electrolytic $K_3Fe(CN)_6$ + NaOH in water. X250.

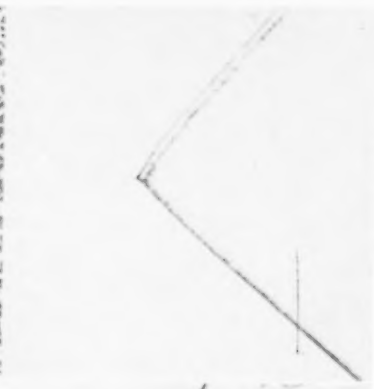

Fig. 4—20.7 pct Mo alloy quenched after annealing at 1480°C. Retained β solid solution. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

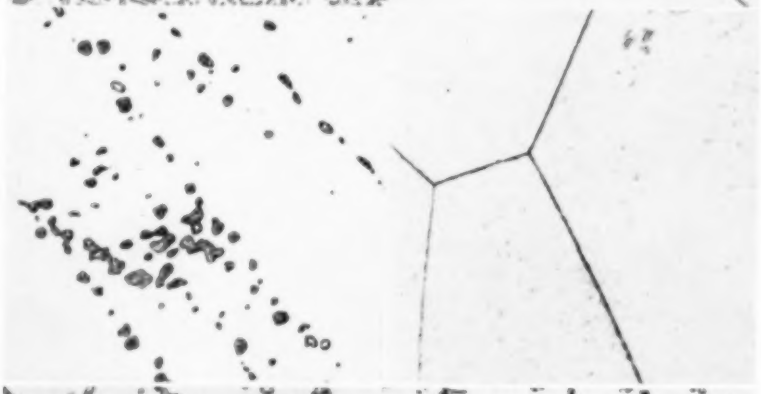

Fig. 5—22.1 pct Mo alloy quenched after annealing at 1480°C. Retained β solid solution plus isothermal $ZrMo_2$ crystals. Etchant: electrolytic $K_3Fe(CN)_6$ + NaOH in water. X250.

Fig. 6—9.6 pct Mo alloy quenched after annealing at 900°C. Retained β solid solution. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

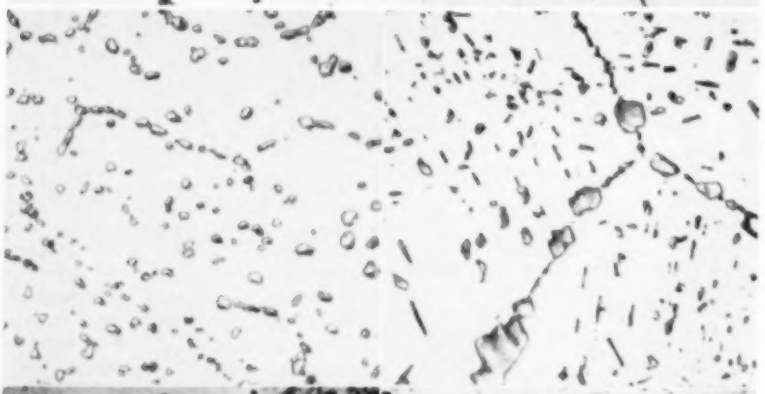

Fig. 7—11.9 pct Mo alloy quenched after annealing at 900°C. Retained β solid solution plus isothermal $ZrMo_2$ crystals. Etchant: electrolytic $K_3Fe(CN)_6$ + NaOH in water. X500.

Fig. 8—14.5 pct Mo alloy quenched after annealing for 600 hr at 790°C. Retained β plus $ZrMo_2$ structure, annealed above eutectoid level. Etchant: electrolytic $K_3Fe(CN)_6$ + NaOH in water. X750.

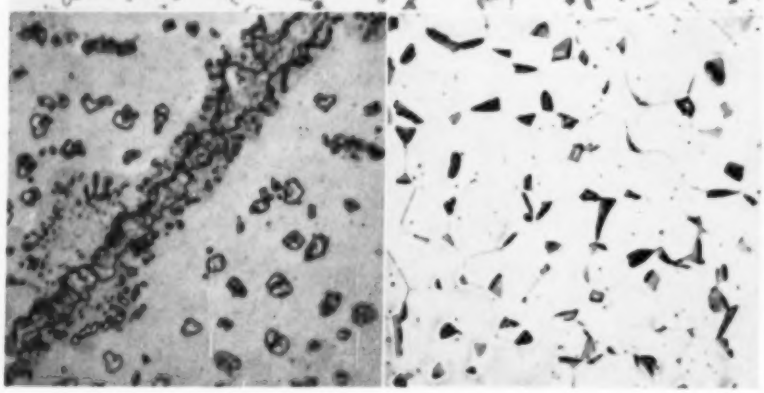

Fig. 9—15.7 pct Mo alloy quenched after annealing for 380 hr at 775°C. A small amount of β adjacent to the proeutectoid $ZrMo_2$ crystals has decomposed into α + $ZrMo_2$. This places the eutectoid level above 775°C. Etchant: electrolytic $K_3Fe(CN)_6$ + NaOH in water. X750.

Fig. 10—0.18 pct Mo alloy quenched after annealing at 775°C. The α + metastable β structure here shows the limited α solubility. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X500.

Gordon,[1] who report $ZrMo_2$ to be cubic, with a = 7.58Å.

A limited amount of work with molybdenum-rich alloys indicated that less than 10 pct Zr is soluble in molybdenum.

Incipient melting and thermal analysis data used in construction of the diagram are given in Table IV.

Zr-W System

The phase relationships in the 0 to 50 pct region are presented in Fig. 13, and the entire diagram is shown in Fig. 14. Most of the experimental points

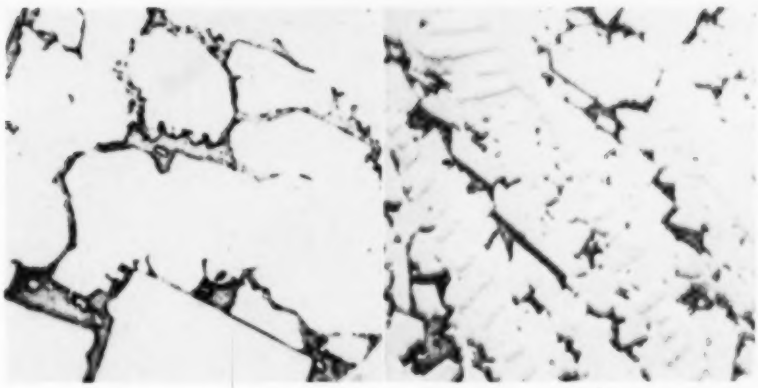

Fig. 11—57.8 pct Mo alloy, as-cast. Primary $ZrMo_2$ crystals plus β solid solution. Etchant: 3 pct HNO_3 + 2 pct HF in water. X750.

Fig. 12—59.0 pct Mo alloy, as-cast. Primary molybdenum-rich solid solution, peritectic walls of $ZrMo_2$, and β matrix. Comparing this with Fig. 11 shows that the composition of the peritectic liquid must lie between 57.8 and 59 pct Mo. Etchant: 3 pct HNO_3 + 2 pct HF in water. X750.

necessary for the construction of the diagram are included in the former figure. The chemical analyses of Zr-W alloys are given in Table V. Table VI lists the annealing treatments employed.

Prior to the isothermal anneals, alloys containing 0.5 to 26 pct W were reduced up to 32 pct by cold pressing, homogenized 45 to 70 hr at 1200°C, and cold rolled up to 20 pct. The amount of reduction by cold working was, of course, dependent upon the wolfram content. The Zr-W system is similar in all structural features to the system Zr-Mo.

The eutectic between β and ZrW_2 was positioned at 18 pct W and 1650°±15°C after examining alloys containing 0.5 to 26 pct W in the as-cast condition and quenched in the incipient melting furnace. The eutectic composition is believed to be accurate within ±2 pct W. The solubility of wolfram in β zirconium decreases from a maximum of 8 pct at the eutectic temperature to about 0.5 pct at the eutectoid temperature, 850°C. Figs. 15 to 18 position the $\beta/\beta + ZrW_2$ phase boundary at two temperature levels. Fig. 15, a 3.6 pct W alloy quenched from 1460°C, contains only transformed β, while a 5.3 pct alloy quenched from the same temperature displays $\beta + ZrW_2$ (Fig. 16). Fig. 17 is a 0.49 pct W alloy quenched from 950°C, illustrating a transformed β structure, and a 1.3 pct W alloy, quenched from the same temperature, Fig. 18, contains ZrW_2 at the boundaries of transformed β grains. No clear retained β structures were observed in this system. Rejection of very fine ZrW_2 particles from β solid solution, even on quenching, masked most structures so that this point was impossible to determine with certainty.

The eutectoid decomposition of β into $\alpha + ZrW_2$ was placed at 850°±15°C. Difficulty was experienced in interpreting some structures because of the low alloy content of the eutectoid (0.5 pct W) and its proximity to the transformation "range" of unalloyed zirconium. Three-phase structures such as that shown in Fig. 19 indicated the proximity to the eutectoid temperature. This is a 1.3 pct W alloy quenched from 850°C.

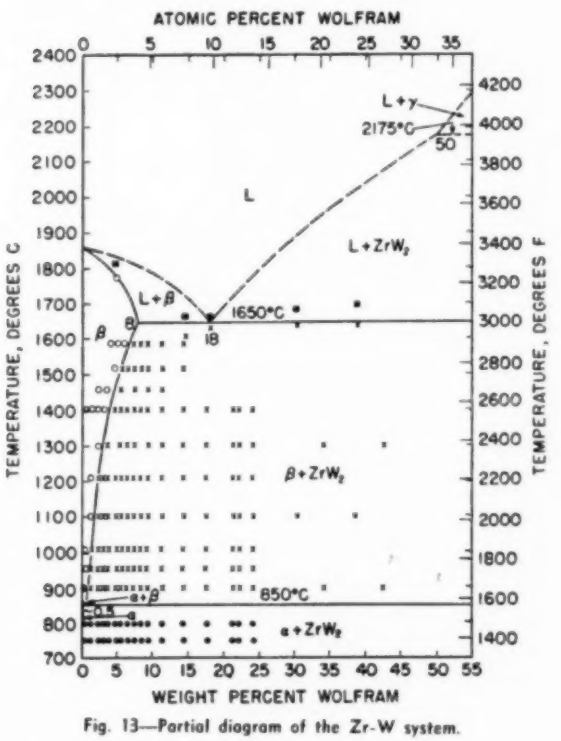

Fig. 13—Partial diagram of the Zr-W system.

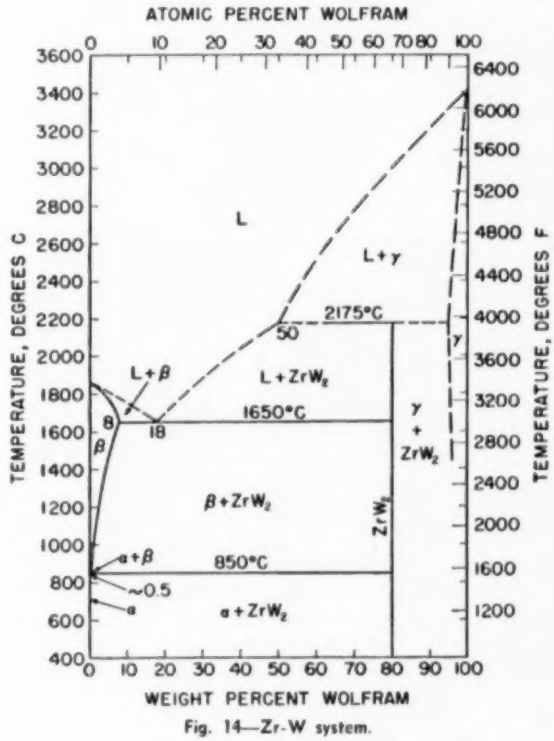

Fig. 14—Zr-W system.

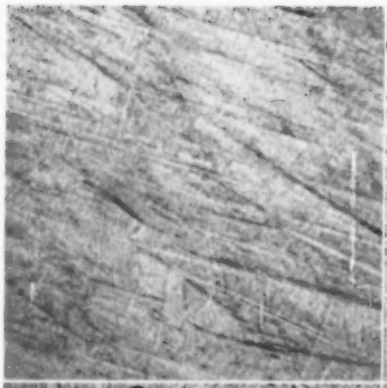

Fig. 15—3.6 pct W alloy quenched after annealing at 1460°C. Transformed β solid solution. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

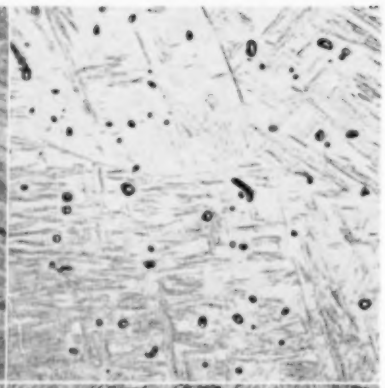

Fig. 16—5.3 pct W alloy quenched after annealing at 1460°C. Transformed β solid solution plus ZrW_2 crystals. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

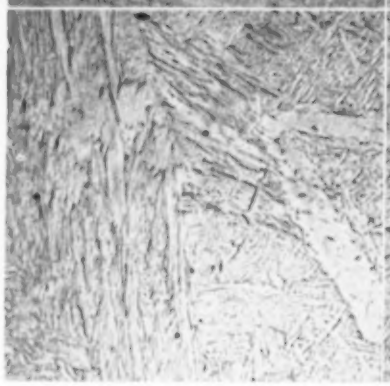

Fig. 17—0.49 pct W alloy quenched after annealing at 950°C. Transformed β solid solution. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

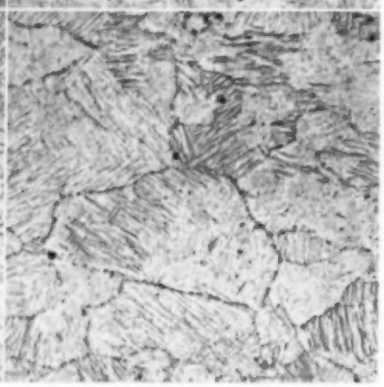

Fig. 18—1.3 pct W alloy quenched after annealing at 950°C. Transformed β solid solution plus ZrW_2 crystals. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X250.

The solubility of wolfram in α zirconium is considerably less than 0.5 pct (the most dilute alloy prepared) at all temperatures. A single intermediate phase, peritectically formed, was detected in this system. Claassen and Burgers[8] have reported this phase to be ZrW_2 (stoichiometrically, 80.13 pct W) having a cubic structure (C15 type) with the lattice parameter, a = 7.61A. Figs. 11 and 12, illustrating the peritectic formation of $ZrMo_2$, are characteristic of the structures observed in a 48.5 and a 52.3 pct W alloy, respectively. The 48.5 pct W alloy contained only ZrW_2 and β phases, while primary dendrites of wolfram-rich solid solution were observed in the latter alloy. Accordingly, the composition of the peritectic melt has been placed at 50 pct W.

Because of the considerable difference in wolfram

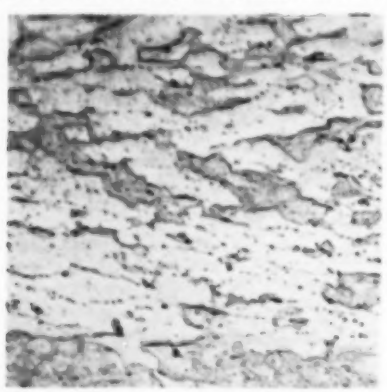

Fig. 19—1.3 pct W alloy quenched after annealing at 850°C. $\alpha + \beta + ZrW_2$ structure characteristic of alloys quenched from this temperature, indicating the proximity to the eutectoid level. Etchant: 20 pct HNO_3 + 20 pct HF in glycerin. X500.

Table V. Analyses of Zr-W Alloys

Alloy Designation	W, Wt Pct	Alloy Designation	W, Wt Pct
ZW 0.5-784	0.49	ZW 25-646	23.5
ZW 1-783	1.3	ZW 26-766	24.0
ZW 2-782	2.2	ZW 30-647	30.4
ZW 3-781	2.9	ZW 35-648	33.9
ZW 4-780	3.6	ZW 40-649	38.5
ZW 5-779	4.7	ZW 45-650	42.6
ZW 6-778	5.3	ZW 46-783	46.6
ZW 7-777	6.1	ZW 49-782	48.5
ZW 8-768	7.2	ZW 50-652	58.0
ZW 9-776	8.2	ZW 52-781	52.3
ZW 10-775	9.3	ZW 55-801	55.4
ZW 12-774	11.3	ZW 60-800	61.3
ZW 14-773	14.0	ZW 65-799	65.5
ZW 15-644	15.2	ZW 70-798	71.3
ZW 16-772	14.5	ZW 75-796	77.9
ZW 18-771	17.5	ZW 80-795	82.8
ZW 20-770	17.9	ZW 85-794	87.6
ZW 22-769	21.1	ZW 90-765	91.2
ZW 24-767	22.2		

Table VI. Schedule of Annealing Treatments for Zr-W Alloys

Annealing Temperature, °C	Annealing Time, Hr	Alloys,* Pct W
1520†	5 min	5-16
1590†	5 min	5-16
1440	0.2	2, 4, ... 26, 30, 40, 50
1400	1	1-26
1300	2	2, 4, ... 26, 35, 45, 55
1204	8	1-26
1100	20	1-26, 30, 40, 50
1006	48	0.5-26
950	50	0.5-26
900	75	0.5-26, 35, 45, 55
850	95	0.5-26
800	170	0.5-26, 30, 40, 50
750	240	0.5-26
700	335	0.5-10, 20, 35, 45, 55
650	450	0.5-10
600	500	0.5-10, 20, 30, 40, 50

* Alloys were pretreated before annealing as described in the text. Control samples of pure zirconium were included in each annealing treatment.

† These treatments were carried out in small molybdenum canisters supported by wolfram wire in the incipient melting furnace.

Table VII. Melting Range Determination for the Zr-W System

Alloy No.	W, Wt Pct	Melting Observed, °C	No Melting, °C
Zr	0	1852	
YZW 5-779	4.7	1815	1775
YZW16-772	14.5	1665	1605
YZW20-770	17.9	1665	1635
YZW30-647	30.4	1695	1645
YZW70-798	71.3	2175	2070
YZW80-795	82.8	2175	2075

content between the peritectic melt and the intermediate phase, and the difficulty in equilibrating the resultant three-phase alloys, nearly single-phase ZrW_2 structures were not obtained. Therefore, X-ray patterns were not made to check the structure of this phase, and the work of Claassen and Burgers has been accepted as valid.

A limited investigation of wolfram-rich alloys indicated that less than 10 pct Zr is soluble in wolfram.

The incipient melting data are presented in Table VII. The results of these studies placed the β-ZrW_2 eutectic temperature at 1650°±15°C and the peritectic at 2175°±25°C.

Summary

The Zr-Mo and Zr-W phase diagrams were carefully determined to 50 atomic pct alloy content. A limited amount of work was done to outline the phase fields in the molybdenum and wolfram-rich regions of the diagrams. Alloys were melted in a nonconsumable electrode furnace using Grade 3 iodide zirconium crystal bar as a base material. These highly reactive alloys were prepared and heat treated under protective atmospheres to prevent contamination.

Zr-Mo: The important features of this system include:

1—Only one intermediate phase, $ZrMo_2$ (ideally 67.78 pct Mo), exists. It is formed by the peritectic reaction: Melt (58 pct Mo) + Mo-rich solid solution → $ZrMo_2$ at 1880°±20°C.

2—A eutectic between β solid solution and $ZrMo_2$ exists at 31±1 pct Mo and 1520°±15°C.

3—The limit of solubility of molybdenum in β zirconium is about 22 pct at the eutectic temperature.

4—A sluggish eutectoid decomposition of β into α + $ZrMo_2$ occurs at 7.5±1 pct Mo and 780°±5°C.

5—The limit of solubility of molybdenum in α zirconium is less than 0.18 pct at all temperatures.

6—Less than 10 pct Zr is soluble in molybdenum.

Zr-W: This system is similar to the Zr-Mo system, and includes the following features:

1—A single intermediate phase, ZrW_2 (ideally 80.13 pct W), exists. It is formed by the reaction: Melt (50 pct W) + W-rich solid solution → ZrW_2 at 2175°±25°C.

2—A eutectic between β solid solution and ZrW_2 occurs at 1650°±15°C and 18 pct W.

3—The limit of solubility of wolfram in β zirconium is about 8 pct at the eutectic temperature.

4—β decomposes into α + ZrW_2 eutectoidally at about 0.5 pct W and 850°±15°C.

5—The solubility of wolfram in α zirconium is less than 0.5 pct at all temperatures.

6—Less than 10 pct Zr is soluble in wolfram.

Acknowledgments

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Technical Note

Sintering of Ultrafine Ferromagnetic Powders

by N. I. Ananthanarayanan and J. F. Libsch

A LITERATURE survey[1-4] of sintering metal powders and powder compacts indicates that studies have so far been confined primarily to the latter stages of sintering in relatively coarse powders. Presumably this is due to the difficult techniques associated with studies of the early stages of sintering. Recent experimental and theoretical investigations[5-9] on the preparation and the magnetic properties of ultrafine ferromagnetic powders suggest the possibility of following the sintering process from a very much earlier stage than has previously been considered.

It is now well-known that ferromagnetic powder particles which are below a critical size behave as single domains and exhibit permanent magnet qualities characterized by a very high coercive force. When the same particles are fully sintered, the coercive force decreases to very low values corresponding to those of the solid metal. Thus the coercive force appears as a sensitive parameter which may be measured to study the early stages of sintering. To determine the feasibility of this technique, experi-

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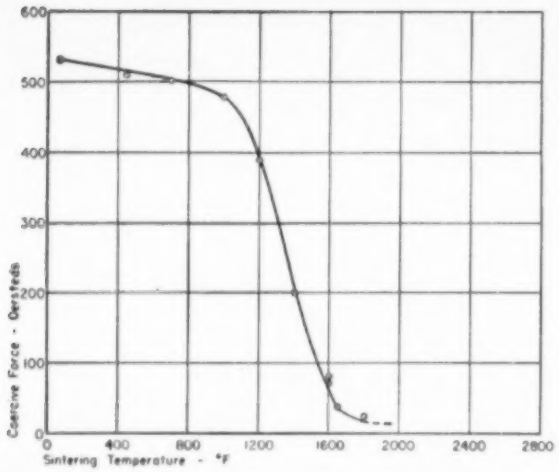

Fig. 1—Variation of coercive force with sintering temperature for ultrafine iron powder compacts.

ments have been initiated on the sintering behavior of ultrafine iron powders.

The present experiments were restricted to powders prepared by the low temperature reduction of finely divided jeweller's rouge, in purified hydrogen. The rouge used contained 98 pct iron oxide and 2 pct SiO_2, and consisted of particles predominantly one micron or less in size. It has been found that of the powders thus prepared in the temperature range of 600° to 1000°F for 200 min of reduction time and for a hydrogen flow of about 1.5 cu ft per hr per g of oxide, the powder reduced at 700°F exhibited the maximum coercive force, viz., 560 to 600 oersteds. This powder was chosen for the experiments reported, because it provided the maximum variation of the coercive force between the "loose" and fully sintered states. Particle size of the reduced powder was estimated by the electron microscope, X-ray diffraction, and nitrogen adsorption methods, yielding an average particle size of 200 to 250 A.U. Since the powders were pyrophoric and were, therefore, covered with liquids like acetone for protection from oxidation during their subsequent handling, they were pressed wet into bars of approximately $1\frac{1}{4} \times \frac{1}{8} \times \frac{1}{8}$ in. at a single pressure of 40 tons per sq in. The compacts, first evacuated in the sintering chamber to remove all traces of protecting liquid present, were sintered in a stream of dry hydrogen for a period of 2 hr at various temperatures between 450° and 1800°F. The coercive force of the sintered compacts was then measured using a high-H permeameter of the Sanford and Bennet type.[10]

The coercive forces of the sintered compacts plotted as a function of sintering temperature are shown in Fig. 1. It may be noted: 1—that up to a temperature of 1000°F there is a very gradual fall of coercive force with sintering temperature; 2—that in the relatively narrow range of temperature between 1000° and 1600°F there is a strikingly rapid diminution of coercive force from about 500 to about 70 oersteds; and 3—that the rate of decrease of the coercive force at the higher temperatures is once again gradual.

The initial slow decrease of coercive force with sintering temperature indicates that little sintering has occurred and that the single domain character of the particles constituting the specimen has been essentially preserved. The relatively narrow range of sintering temperature in which the coercive force undergoes an abrupt diminution marks the excessive formation of domain boundaries between the individual particles by sintering. The subsequent slow decrease of the coercive force with increasing sintering temperatures is to be associated with elimination of the porosity, the slow progress of which, as is well-known, is characteristic of the latter stages of sintering. The final value of the coercive force reported at the highest sintering temperature used, e.g., 25 oersteds at 1800°F, is still high compared with that of massive iron, because of the presence of impurities and the low density obtained, e.g., approximately 4 g per cc.

Any interpretation of the data in terms of the sintering mechanism must recognize the presence of some SiO_2 and some oxygen in the pressed compacts before sintering, since the SiO_2 was present in the rouge used for reduction and reduction of the oxide is not complete at 700°F. The presence of these impurities may have a pronounced influence and would tend to retard sintering by interfering with the formation and lateral growth of point bonds. With pure iron powders completely reduced, the temperature range of sudden decrease in coercive force would be expected to shift to the left.

From the data presented, it appears that the temperature range representing a sudden decrease in coercive force may be an indication of appreciable sintering and the consequent formation of excessive domain boundaries. While the initial and final values of the coercive force may depend chiefly upon the particle size, the purity and possibly the porosity of the powder particles, it is believed the temperature range of sudden decrease in coercive force will depend primarily upon the factors which influence the initiation and progress of sintering. A shift in this temperature range may conceivably be used to show the influence of various nonmetallic films on the powder particles, pressing pressure, and sintering atmosphere upon the initiation and progress of sintering. Experiments to determine the usefulness of this new criterion are presently underway.

The use of coercive force as a parameter for sintering behavior is, of course, limited to ferromagnetic materials. However the electrical resistivity of metals and alloys, another structure-sensitive property, is also known to undergo a large change below a critical size, as evidenced by the many and scattered investigations that have been reported on thin metal films.[11] The measurement of electrical resistivity might perhaps form a still more general approach to the study of building up polycrystalline aggregates from ultrafine powders by the powder metallurgy technique.

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Measurement of Internal Boundaries in Three-Dimensional Structures By Random Sectioning

by Cyril Stanley Smith and Lester Guttman

It is shown, from a study of geometric probabilities, that the average number of intercepts per unit length of a random line drawn through a three-dimensional structure is exactly half the true ratio of surface to volume. Since the surfaces can be internal or external, the area of grain boundary or of the interface between any two constituents in a microstructure can be measured. Other metric relations are tabulated that may be of use in studies of the microstructure of polycrystalline, cellular, or particulate matter generally.

IN many fields of scientific investigation the structure of cellular aggregates or random arrays of discrete particles imbedded in some matrix is observed on a two-dimensional section and inferences are drawn therefrom as to the real structure in three dimensions. The biologist's microtome slice, the petrologist's thin section, and the metallurgist's plane polished and etched sections are common examples, although the problem is a general one. Scientists have commonly limited their thinking to the same dimensionality as their structures, and the few attempts that have been strictly three-dimensional in character have been laborious and noteworthy.

From a metallurgical standpoint it is often of considerable importance to know, in addition to the volume fraction of two or more components in an alloy, the amount of two-dimensional interface between crystals. Such grain boundaries (which may separate either two identical crystals differing only in orientation or two crystals differing in structure, and possibly also in orientation) have a determining factor upon the mechanical behavior. It is at these boundaries that melting commences, that stress-induced corrosion occurs, and that various precipitates (harmful or otherwise) first appear. The boundary is doubtless of equal importance in nonmetallic crystalline aggregates such as rocks, ceramics, and concrete, and the biologist is deeply concerned with the area of cellular membranes. Many synthetic cellular foams involve similar structural problems. The very term structure usually implies the presence of interfaces and a complete understanding of structure involves nothing but an analysis of the geometrical, metrical, and topological relations between the various interfaces (zero, one and two-dimensional) that exist in a three-dimensional structure. Even systems lacking sharp physical interfaces often have interrelated gradients of composition or velocity (as cored crystals or turbulence cells) in which a neutral surface can be treated as a two-dimensional interface.

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In an earlier paper by one of the authors[1] the question of cell shape was considered in terms of simple topological principles without regard to physical dimensions. The determination of the actual size of grains in two dimensions is carried out in a routine fashion in innumerable metallurgical laboratories (see, for example, the ASTM standard methods of grain size determination[2]), though this is done merely to check the uniformity of a product and has no relation to the actual three-dimensional shape or size of the grains. Some authors have discussed the three-dimensional problem but only on the basis of assumptions as to idealized grain shapes.[3-5] Quantitative measurements of microstructures to obtain the volumetric relations of various phases have been carried out by petrographers for many years and are of increasing popularity among metallurgists.[6]

The present paper will show how, on the basis of no assumptions other than randomness of sectioning (usually realizable in experiment), it is possible to learn a great deal about the three-dimensional structure. The relations to be derived will generally be used on random arrays of cells or other particles, although they are equally applicable to ordered arrays and even to isolated objects of complex shape provided that suitable random sections can be made.

Total Area of Interfaces in a Sample

Consider a typical microstructure of a single-phase polycrystalline metal, such as that shown schematically in Fig. 1. The plane cross section shown contains a network of lines which subdivides the area into two-dimensional cells. The lines, of course, represent merely the intersection of the two-dimensional plane of sectioning with the two-dimensional interfaces between adjacent three-dimensional cells. The structure also contains points at which two or more lines intersect; in three dimensions the points are, of course, lines.

In the more general case there may be in three dimensions isolated particles surrounded by a single interface without contact with others, and both two and one-dimensional features which do not necessarily connect with others. On the two-dimensional section these will appear as areas delineated by a closed line (as at *a* and *b*, Fig. 2), as isolated lines

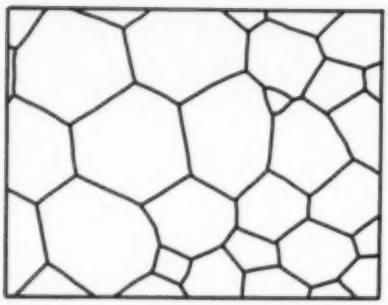
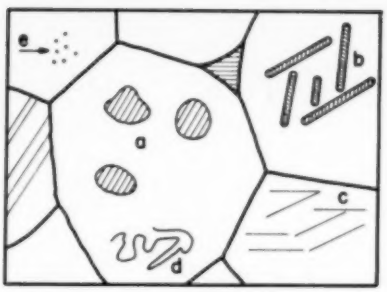


Fig. 1 (left)—Plane section through a polycrystalline body.

Fig. 2 (right)—Plane section through a polycrystalline body containing isolated particles (sectioned at *a* and *b*), isolated surfaces (sections *c* and d), and lines sectioned at *e*.



not intersecting others (as at c and *d*), or just as isolated points (*e*).

Relations in Two Dimensions: The two-dimensional structure itself will be considered first. It is possible by the use of a map measurer to determine the total length of line in any given area isolated for study. However, this can be done more easily by making use of the probability of intersection of one line with another randomly applied. It is intuitively apparent that the number of intersections will depend in a simple manner on the length of line, and that there will exist a relationship between an area and the length of random line traversing it. By combining these relations it is possible to obtain the ratio of line to area as a simple direct function of the average number of intercepts per unit length of an intersecting straight line (or grid) randomly applied to the structure.

We begin by rederiving the well-known solution to Buffon's "needle" problem:* a line segment of length l falls at random on a plane array of parallel lines spaced a distance d apart (Fig. 3). What is the probability that the line segment intersects one of the lines ($l \leq d$)?

The probability is clearly the average length of the segment projected on the normal to the array, divided by the distance between the lines, i.e.:

$$p = \frac{\bar{l}_{proj}}{d}$$

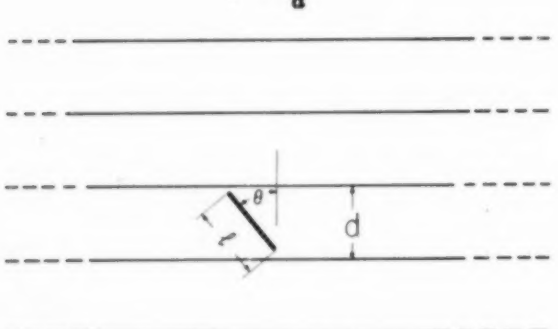


Fig. 3—Diagram to illustrate probability of a line intersecting a grid.

Since all directions of the line segment have equal *a priori* probability:

$$\bar{l}_{proj} = \frac{2}{\pi}\int_0^{\pi/2} l \cos\theta \, d\theta = \frac{2l}{\pi} \qquad [1]^{**}$$

and:

$$p = \frac{2l}{\pi d} \qquad [2]$$

If an irregular plane curve is placed at random on the same array, there will in general be N intersections; the average value, $\bar{N}$, can be obtained from Eq. 2 as follows. Divide the curve into short segments of length Δl, each of which is nearly straight. The probability that any one of these segments intersects the array is $p_i = 2\Delta l/\pi d$, and the product of this with the number of segments is the average number of intersections:

$$\bar{N} = p_i \cdot \frac{l}{\Delta l} = \frac{2l}{\pi d} \qquad [3]$$

l is the total length of the curve. Even though the segments are connected by being parts of the curve, each takes random positions and orientations as the curve as a whole does, hence the additivity of the number of intersections.

In dealing with a given area of a microstructure it is often useful to express the results in terms of ratio of length to area. Consider the limited area*** in Fig. 4 crossed by a grid of spacing d. If d is small, or if a coarse grid is applied many times at random to a fixed area, the total length of grid line, L, is A/d. Combining with Eq. 3, the following useful relation results:

$$\frac{l}{A} = \frac{\pi}{2}\frac{\bar{N}}{L} \qquad [4]$$

In using this relation it is not necessary to utilize a well-ruled grid of parallel lines, but any array of lines repeatedly applied to the structure in a random fashion is satisfactory provided that a sufficient number of intersections is counted. If the structure itself is random, a single line (not necessarily straight) of sufficient length is adequate. Generally, however, it is preferable to use a grid and to apply this successively at various angles between 0° and

* Czuber (ref. 7) pp. 84-92. None of the other relations derived in the equations to be given are to be found in their most general forms in the standard works on geometrical probability which have been consulted. Czuber's theorem III (p. 113), IV (p. 115), Problem XXI (p. 116) and Problem III (p. 178) contain formulas similar to ours, but are essentially restricted to convex plane curves. Czuber apparently did not realize that the concept "probability of intersection" could be generalized to "average number of intersections," and thus apply to non-convex curves.

Note added Sept. 30, 1952: After this paper was submitted, there appeared a communication by Campbell and Tomkeieff (*Nature* (1952) **170**, p. 117) containing results similar to ours, in which, however, the unnecessary restriction to convex bodies is still made. L. A. Santaló has derived two-dimensional formulas like ours and has stated that the extensions to three dimensions are obvious. *Cf.* (a) *Abh. Math. Sem.* Hansischen Univ. (1940) **13**, pp. 284-294; *Math. Reviews* (1941) **2**, p. 13. (b) *Revista Union Mat. Argentina*, (1943) **9**, pp. 145-164; *Math. Reviews* (1947) **8**, p. 170.

** From this follows an interesting property of all simple plane figures that are not concave. Since each element of the perimeter appears twice within the average projected width, the latter must be exactly $1/\pi$ of the perimeter. It is also to be noted that the average linear intercept across such a figure is $\pi \times$ area/perimeter.

*** This figure intentionally contains features impossible in any real microstructure to emphasize the independence of the relations on anything but the existence of one and two-dimensional features.

180° selected to give the desired total number of counts.

There are no limitations whatever as to the validity of Eq. 4 except for the critically important requirement of randomness.

Relations in Three Dimensions: Consider now the relation between a random two-dimensional section and the three-dimensional structure it represents. Fig. 5 depicts an irregular solid body with closed surface, intersected by a set of parallel planes. This is analogous to the two-dimensional case of Fig. 3. The sections visible in the two-dimensional planes will have an area A and a perimeter of length l. The averages of these, $\bar{A}$ and $\bar{l}$, are desired for all possible positions of the body. Let the distance between the planes again be d, and assume for simplicity that the surface does not intersect more than one plane at a time. The position of the surface can be specified by the distance z of a fixed point on it from one of the planes, and its orientation by three

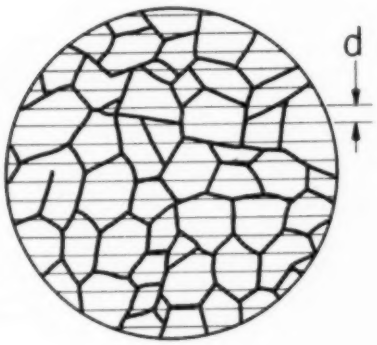


Fig. 4 — Isolated area of a two-dimensional structure traversed by a grid of lines.

angles, denoted by ω for brevity. The area of the section is a function $A(\omega, z)$ whose average value is:

$$\bar{A} = \frac{\int d\omega \int dz\, A(\omega, z)}{\int d\omega \int dz}$$

For a fixed orientation, $A(\omega, z)dz$ is the element of volume of the solid enclosed by the surface. Hence as z varies between the limits $(0, d)$,

$$\int_0^d dz\, A(\omega, z) = V$$

a result independent of orientation.

$$\bar{A} = \frac{V}{d} \qquad [5]$$

There is no restriction on the shape of the surface or on the connectivity of the solid enclosed by it: The distinction between "inside" and "outside" determines what is to be counted as contributing to the cross-sectional area, and conversely.

Consider now a plane figure which intersects one of the planes of the stack in a line of length l (Fig. 6). Again, the average length of intersection is:

$$\bar{l} = \frac{\int d\omega \int dz\, l(\omega, z)}{\int d\omega \int dz}$$

For a fixed orientation, $l\,dz$ is just the element of surface of the figure, projected on a plane which includes the intersection and which is normal to the stack of planes, i.e.:

$$l\,dz = dS \sin\theta$$

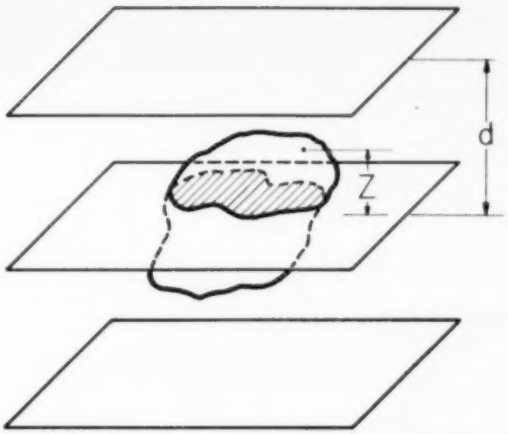


Fig. 5—Diagram to illustrate probable area of intersection of a particle with a set of parallel planes.

where θ is the angle between the normal to the figure and the normal to the stack. Hence, the integration over z gives just the total surface area projected on this vertical plane. The only orientation angle ω which must be specified is θ, and:

$$d\omega = \sin\theta\, d\theta$$

The range $(0, \pi/2)$ for θ clearly covers all possible orientations, so that:

$$\bar{l} = \frac{S \int_0^{\pi/2} \sin^2\theta\, d\theta}{\int_0^{\pi/2} \sin\theta\, d\theta \int_0^d dz} = \frac{\pi S}{4d} \qquad [6]$$

This result is not restricted to plane figures, since any surface can be approximated by plane elements. As before, these elements take random positions and orientations when the figure as a whole does so, and since the contributions to the surface and to the length of intersection are additive, Eq. 6 holds for any surface, however complex.

By combining Eqs. 5 and 6:

$$\frac{\bar{l}}{\bar{A}} = \frac{\pi}{4}\frac{S}{V} \qquad [7]$$

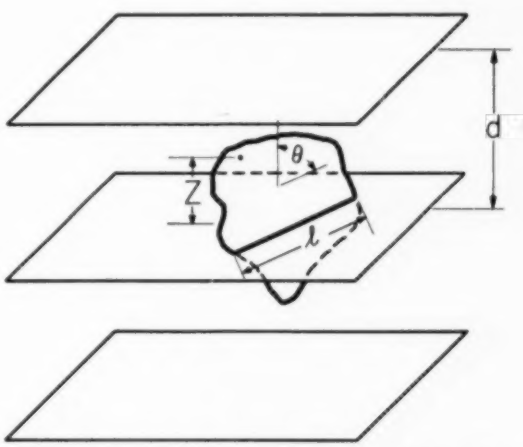


Fig. 6—Diagram illustrating probable length of intersection of a plane figure with a set of parallel planes.

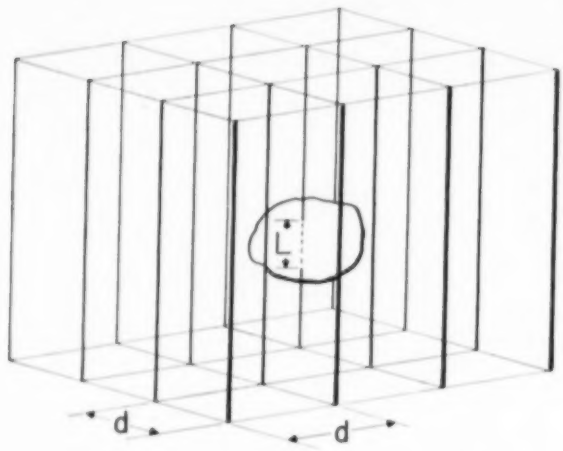


Fig. 7—Intersection of grid of lines with a particle.

This result, being independent of d, means that the stack of parallel planes is no longer needed, and that it is possible to average line length and area of the sections of any solid by a plane oriented and placed at random.

It is desired to apply Eq. 7 to a solid such as a polycrystalline metal, in which the interest is specifically in the internal surfaces (grain boundaries). If the solid is macroscopically homogeneous, the quantity S/V will be a "property" of the specimen in the sense of having the same value for sufficiently large regions, whatever their location. Then it will be possible to obtain its value for the entire specimen from measurements of the average value, $\bar{l}$, of line length in many equal areas, A, randomly located. If the solid is isotropic as well, a single section will suffice if it is extensive enough, but in general both random directions and random locations of the sections would have to be established.

It is not necessary to measure $\bar{l}$ directly, since Eq. 4 provides a means of determining the length of an irregular curve by random intersections with another of known length. Since the $\bar{A}$ and $\bar{l}$ of Eq. 7 become the A and l of Eq. 4, then

$$\frac{S}{V} = \frac{2\bar{N}}{L} \qquad [8]$$

Note that d does not appear in this equation. It means simply that the internal surface in a unit volume of a solid is exactly twice the average number of intersections per unit length of lines drawn at random in the solid.

Eq. 8 can be deduced more directly in another way. Consider a grid of parallel lines spaced a distance d apart in directions normal to each other and to the lines. An irregular solid is placed in the grid (Fig. 7); the average number, $\bar{N}$, of intersections of its surface with the lines and the average length of line intercepted, $\bar{L}'$, are desired. The position and orientation of the solid are described by the coordinates (x, y) of one of its points in the plane perpendicular to the lines, and by a set of angles, ω, as before. Then:

$$\bar{L}' = \frac{\int d\omega \int dx \int dy\, L'(x, y, \omega)}{\int d\omega \int dx \int dy}$$

For fixed ω, $L'\,dx\,dy$ is the volume element, and $\iint L'\,dx\,dy = V$, independent of angle. Hence:

$$\bar{L}' = \frac{V}{d^2} \qquad [9]$$

a result which is intuitively obvious. For a plane figure, whose largest diameter is $\leq d$, the probability of intersection is just the ratio of its average projected area to the area per grid line, i.e.:

$$p = \frac{\bar{A}_{proj}}{d^2}$$

But:

$$A_{proj} = S \cos\theta$$

and:

$$\bar{A}_{proj} = \frac{\int_0^{\pi/2} S \cos\theta \sin\theta\, d\theta}{\int_0^{\pi/2} \sin\theta\, d\theta} = \frac{S}{2} \qquad [10]\dagger$$

† From Eq. 10 a result of interest for non-concave solids in general can be deduced; on the average, each element of the total surface, S, of such a solid appears twice within the same projected area, so that, for convex solids only:

$$\bar{A}_{proj} = \frac{S}{4} \qquad [10a]$$

This theorem has been attributed to Cauchy, but the original publication has not been located.

whence:

$$p = \frac{S}{2d^2} \qquad [11]$$

Table I. Properties of a Two-Dimensional Structure

Item	Equation No. in Text	Property	Contiguous Cells of Same Type	Separate Cells Not Sharing Boundaries
1		Average area of cell	$\frac{A}{M}$	$\frac{L_a d}{M_a}$
2		Average linear intercept	$\frac{L}{N}$	$\frac{2L_a}{N_a}$
3	4	Ratio of length of line to total area (perimeter to particle area in separate cells)	$\frac{\pi N}{2L}$	$\frac{\pi N_a}{2L_a}$
4		Ratio of average perimeter of cell to average linear intercept*	$\pi \frac{N^2 A}{ML^2}$	$\frac{\pi N_a^2 d}{4M_a L_a}$
5		Area fraction of phase α		$\frac{L_a}{L}$

* This dimensionless shape factor is rigorously applicable only if all cells are convex and if there are no lines except those constituting the perimeter of some cell. It is 4 for circles, $24\sqrt{3}\pi$ for regular hexagons, $16/\pi$ for squares, and $36/\pi\sqrt{3}$ for equilateral triangles. (See double asterisk note to Eq. 1.)

Table II. Properties Determinable Rigorously from Random Two-Dimensional Sections of Three-Dimensional Structures

Item	Equation No. in Text	Property	Contiguous Cells	Separate Cells or Particles Not Sharing Boundaries and with No Internal Features
1		Average cross section of cell or particle	$\frac{A}{M}$	$\frac{L_\alpha d}{M_\alpha}$
2		Average linear intercept	$\frac{L}{N}$	$\frac{2L_\alpha}{N_\alpha}$
3	7, 8, 13	Ratio of area of total two-dimensional features to total volume (surface to volume ratio for separate particles)	$\frac{4l}{\pi A}$ or $\frac{2N}{L}$	$\frac{2N_\alpha}{L_\alpha}$
4	14	Ratio of length of total one-dimensional features to total volume (edge to volume ratio for separate particles)	$\frac{2n}{A}$	$\frac{2n}{L_\alpha d}$
5	15	Ratio of total one-dimensional to total two-dimensional features (edge to surface ratio for separate particles)	$\frac{\pi n}{2l}$ or $\frac{nL}{NA}$	$\frac{n}{N_\alpha d}$
6		Volume fraction of particles of α phase		$\frac{L_\alpha}{L}$

A = total area of two-dimensional section examined.
l = total length of linear features in area A.
M = total number of grains, cells or particles seen in area A.
n = total number of points (grain corners) seen in area A.
M_α = number of separate particles of constituent α.
L_α = total length of line traversing area occupied by constituent α.
L = total length of line in grid of area A applied to structure.
d = spacing of lines in grid.
N = number of intercepts of grid lines with linear features of structure.
N_α = number of intercepts of grid lines with lines bounding constituent α.

More generally, for any surface:

$$\overline{N} = \frac{S}{2d'} \qquad [12]$$

S being the total surface area, including internal or re-entrant positions. As before, this follows from the possibility of building any surface from plane elements, each of which takes up random positions and orientations as the surface as a whole does so, and to each of which Eq. 11 applies.

Combining Eqs. 9 and 12, then

$$\frac{\overline{N}}{\overline{L'}} = \frac{S}{2V} \qquad [13]$$

The grid is no longer necessary, and if, as for Eq. 8, the number of intersections with random lines of total length L is measured, it may be seen that Eq. 13 leads to the same result.

The three-dimensional structure also may have one-dimensional cells (lines) either defining the limits of various two-dimensional surfaces or existing by themselves without any relation to such. The number of intersections, $\overline{n}$, with a set of planes d apart (randomly applied) can easily be shown by the methods used in deriving Eqs. 3 and 5 to be $\lambda/2d$, λ being the true length of the line. Since the total area of the intersecting planes and the volume considered are related as before,

$$\overline{n} = \frac{\lambda A}{2V} \quad \text{or} \quad \frac{\overline{n}}{A} = \frac{\lambda}{2V} \qquad [14]$$

This gives directly by the observation of points in random plane sections the length of line in a given volume. This relation can be combined with Eq. 2 dealing with the ratio of line to surface to give directly (on the basis of purely two-dimensional measurements) the ratio of line to surface, which is:

$$\frac{\lambda}{S} = \frac{\pi}{2}\frac{\overline{n}}{l} = \frac{\overline{n}}{A}\frac{L}{N} \qquad [15]$$

Use of the Relations

It should be noted that in all of the preceding, L, A, and V are gross values determined by the area

Table III. Properties Determinable on Assumption That Particles Are of One Size and Spherical

Item	Equation No. in Text	Property	Contiguous Cells of Same Type	Separate Particles Not Sharing Boundary
1	16	Average volume of grain or particle	$\frac{4NA^2}{\pi LM^2}$	$\frac{2N_\alpha A^2}{\pi M_\alpha^2 L}$ or $\frac{2N_\alpha}{\pi M_\alpha^2} Ld^2$
2	16	Number of particles per unit volume of whole sample	$\frac{\pi LM^2}{4NA^2}$	$\frac{\pi LM_\alpha^2}{2N_\alpha A^2}$ or $\frac{\pi M_\alpha^2}{2N_\alpha Ld^2}$
3		Average surface area per grain or particle	$\frac{8}{\pi}\left(\frac{NA}{LM}\right)^2$ (See note*)	$\frac{8}{\pi}\left(\frac{N_\alpha d}{M_\alpha}\right)^2$

* Note: If the average surface area of the grains considered as separate polyhedra is desired, this number must be doubled.

selected for study, and the mere counting of intersections within this area gives the total amount of surface and of line. There are no assumptions whatever except that of randomness of sectioning. If the structure itself is random and isotropic a single surface—or even a single line—will suffice provided only that the number of counts is large enough to give satisfactory statistics; if the structure is anisotropic, true averages will only be obtained by combining counts on sections which are randomly oriented and placed.

The factor N/L is the reciprocal of the average linear intercept through the cells assuming them to be convex. Though metallurgists have frequently used this average linear intercept as an index of grain diameter, the fact that it is so simply and exactly related to the ratio of surface to volume seems not to have been previously noticed.

It is possible to experimentally measure on an actual two-dimensional section the total number of intercepts, N, with grain boundaries of a traversing line of a known total length, L (the total being made up of many lines, a distance d apart, repeatedly crossing the area being examined††) as in Fig. 4; the number, M, of particles appearing in section on the whole surface of known area, A; the number, n, of points ("grain corners") seen in a section. If more than one phase is present, it is possible to measure for each phase the number of intercepts (N_α) of the traversing line with its boundaries, the total length of line (L_α) traversing that phase, and the number (M_α) of α-phase particles seen on an area A.

The results of the preceding paragraphs have been rewritten consistently in terms of the quantities just defined and collected in Tables I and II. The tables also include immediately derivable expressions for the cross-sectional area of a particle or cell, A/M, and the average linear intercept through a particle, L/N. Two sets of formulas must be used, corresponding to the case of one phase or of separate phases. In the latter case, in order to study each phase separately, the actual area of each, $L_\alpha d$, must be used instead of A, and the linear intercept becomes $2L_\alpha/N_\alpha$. If there is more than one phase present the volume percentage of any given phase is precisely determined by the ratio L_α/L_{total} in the now well-known method of linear analysis.[9]

†† In practice d is either the spacing of a grid of parallel lines used for counting or the total area studied divided by the total length of traversing line (i.e., $d = A/L$). Experimentally, it is convenient to use a grid of circular outline which can be applied repeatedly at different orientations to portions of an extended microstructure. The value of A/L is then constant and needs no further measurement. If the specimen area itself is limited and falls always entirely with the grid, its true area is used, and the equivalent L derived from the grid spacing. Counting is easier if, instead of a fine grid in a few orientations, a grid is used of fairly coarse spacing, applied frequently and in many orientations in order to get the necessary counts. To avoid repetition at 180° rotation the grid lines should be displaced from exact symmetry or the grid itself should be moved around randomly within the area available for study. A grid 20 cm diam with lines 1 cm apart has been found satisfactory. With structures on the scale normally used for an 8x10 in. micrograph 12 applications 15° apart give an adequate number of counts.

The counting of intersections or cells is most easily recorded by a counting pencil of the type used by bacteriologists, and may be either electrically or mechanically operated. Instead of using a photograph, measurements may be also made directly on a specimen under the microscope, the grid being either projected optically upon the sample or being laid on the focusing screen of a projection apparatus. A large specimen area may easily be covered in this way by appropriate traversing at properly distributed angles. It would be possible to use a counter operated by a photocell and to record all necessary data automatically, provided that clearly defined boundaries are obtainable and there are no extraneous details in the specimen that would affect local light intensities.

The measurement of the length of linear intercept is most readily obtained by the use of a linear integrating stage of the type used by petrographers, such as that of Hurlburt,[8] or Wentworth,[9] or the hydraulic one designed by one of the authors.[10] It is possible to replace these rather elaborate devices by a simple inexpensive stage for point-counting,[11] but this seems to be both more tedious in operation and, unless an extremely large number of points are counted, less accurate.

Shape-Dependent Properties

In all of the foregoing it was not necessary to make any assumptions as to the form of the structures under consideration. Therefore very general results were obtained which are true for all shapes, but which cannot for just that reason give information about shape-dependent properties. In this class of properties are the average "caliper diameter" and the average projected area of a particle, neither of which can be measured on a cross section. In this class also, therefore, is the particle volume, which is equal either to the product of the caliper diameter in a certain direction and the average sectional area normal to it, or to the product of the area projected on a certain plane and the average linear intercept in the perpendicular direction.

For the special case of a spherical particle there are simple relations between those properties that are accessible from a plane section and those that are not. If a random arrangement of identical spheres is sectioned, the number of spheres P_α in a volume V can be found from:

$$\frac{P_\alpha}{V} = \frac{\pi}{2}\left(\frac{L}{N_\alpha}\right)\left(\frac{M_\alpha}{A}\right)^2$$

in the notation of the previous section.‡ It may be assumed that this formula holds approximately for grains which are not too elongated in any particular direction, and which fill space, and thence the volume per particle, V_p, as the reciprocal of the above, may be calculated:

$$V_p = \frac{4}{\pi}\left(\frac{N}{L}\right)\left(\frac{A}{M}\right)^2 \qquad [16]$$

the factor two coming from the sharing of boundaries in the present case. By combining Eq. 16 with earlier results, the formulas appearing in Table III are obtained. It should be stressed that these formulas, unlike those in Tables I and II, must be systematically in error, and that they should be used with caution.

‡ Relations identical with these have been independently derived by both F. N. Rhines and R. Teitel. These authors discuss the use of these relations in determining grain size or particle size and the distribution of these. Apparently the effect of departures from the rigorous requirement of sphericity is not serious in practice.

Summary

The extent of one and two-dimensional internal boundaries of a solid (e.g., grain edge length and grain boundary area in a polycrystalline metal) can be calculated from measurements on plane sections.

The method involves counting the number of intersections of the boundary with planes of known area or lines of known length oriented and located at random. For example, if a line randomly intersects an array of surfaces or interfaces, then the average number of intersections per unit length is exactly equal to one-half the ratio of surface area to volume. The accuracy of the results is only statistically limited and does not depend on assumptions of shape or isotropic arrangement. True randomness of section is essential.

It is possible to obtain the length or area of a particular kind of feature, for example, the area of interface between any two phases, as well as the surface to volume ratio for any constituent, in addition to the total interface measurements.

It is not possible to derive the volume of the average cell from measurements on two-dimensional sections except on the assumption of a constant shape

and size. Formulas derived for identical spheres are tabulated and may be of some use.

Acknowledgment

The authors wish to express their thanks to the industrial sponsors of the Institute for the Study of Metals for the financial support that has made this work possible.

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Technical Note

Retained Austenite Determinations by X-Ray Methods

by B. L. Averbach

THE determination of retained austenite by X-ray diffraction uses the following relationship:[1,2]

$$P_\alpha = \text{constant} \cdot R V_\alpha A(\theta) \qquad [1]$$

where: P is the diffracted power from phase α; R, the calculated intensity factor; $A(\theta)$, the absorption correction; θ, the diffraction angle; and V_α, the irradiated volume of phase α

For a flat sample making a glancing angle ϕ with the incident beam the absorption correction is calculated as:*

$$A(\theta) = \frac{1}{\mu} \frac{\sin(2\theta - \phi)}{\sin(2\theta - \phi) + \sin\phi} \qquad [2]$$

where μ is the linear absorption coefficient.

Beu[5] has proposed that the computed absorption correction be combined with the constant and that the experimental observation of the absorption correction used in the original method be eliminated. This procedure is permissible only if: 1—There is no preferred orientation in the sample, and 2—The geometric requirements have been met precisely.

If these conditions are not met the validity of the determination is questionable. Unfortunately the necessary conditions must be tested experimentally for each determination, and this is done most easily by observing whether the apparent absorption has the form of Eq. 2. In practice it may be convenient to plot P/R vs the parameter $\sin(2\theta - \phi)/[\sin(2\theta - \phi) + \sin\phi]$ to obtain straight lines for the apparent absorption correction. Deviations from straight lines indicate either that the sample has not been positioned correctly or that the relative intensities are incorrect.

Geiger counter spectrometer methods also have been used successfully for retained austenite determinations. In the usual spectrometer arrangement the sample makes equal angles with the incident and diffracted beams ($\theta = \phi$); thus the absorption correction is simply $\frac{1}{2}\mu$ and independent of θ. The retained austenite is then easily calculated from integrated intensity measurements on austenite and martensite lines. If the austenite content is not too low and if excessive fluorescence is not encountered it is possible to use filtered radiation and a recording spectrometer. The integrated intensities are then proportional to the areas of the diffraction peaks.

For lower austenite contents the following technique was used: A bent quartz monochromator and a Geiger counter spectrometer are employed. The monochromatic radiation is focused on the front slit, and the counter slit is made broad enough to include the entire line. A series of readings are taken in the vicinity of the diffraction peak. The highest reading includes the diffraction line plus background. The background is obtained from the average of several readings on each side of the peak. The integrated intensity is obtained by subtraction. CrK_α and FeK_α have been used for such determinations. This technique has been found to be as sensitive and as accurate as the photographic methods.

In the spectrometer methods the ratio austenite/martensite may be obtained from readings on austenite and martensite lines and the application of Eq. 1. It is also possible to use external standards and to obtain the austenite contents from measurements on the austenite lines alone. The small correction for carbides then becomes unnecessary. The best standards are probably samples with known

* The absorption correction in ref. 1 erroneously contained an extra $\sin\phi$ in the numerator. This error did not influence the results quoted since experimental absorption corrections were used in the retained austenite determinations.

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austenite contents although α brass and rock salt have also been used.

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Technical Note

Regarding Sigma Phase Formation

by David S. Bloom and Nicholas J. Grant

IN recent reports, Sully[1] and Beck and coworkers[2] have advanced hypotheses concerning the formation of the σ phase. Both of these hypotheses are based on Pauling's theories of the electronic configuration of the elements of the first transition group. Sully considered that the number of electrons which can be absorbed in filling electron "holes" is the factor which determines whether or not the σ structure can form. Beck suggested that the presence of a certain concentration of electron "holes" is the controlling value.

It can be shown that a similar criterion can be developed without making reference to Pauling's theories. Table I shows the elements of the first transition group and the incidence of the σ structure in the binary systems of these elements according to the data presently available. Also appended is the number of electrons considered to exist in the 3*d*-4*s* levels of these elements. If the atomic percentages for the σ phase are listed as shown in Table II, and if the number of electrons in the 3*d*-4*s* levels per atom is calculated for the σ boundary values, the results are as shown in the last column. It can be seen that the numerical values are all in the vicinity of 7 electrons per atom. This is true also for the ternary Cr-Mo-Ni σ phase, which occurs in a system in which none of the binary compositions are known to develop the σ phase. In the Fe-Mo binary, the high temperature σ phase, which occurs at a composition of 50 atomic pct of each element, also yields a value of 7 electrons per atom.

From inspection of Tables I and II, it develops that any series of sequential numbers which is assigned to the elements under consideration will result in the numbers pertaining to σ compositions grouping themselves around one specific number. It does not follow, however, that this random number necessarily has physical significance. Such a point has already been discussed by Hume-Rothery.[3]

One interesting point develops from this study, however, and that is the position of manganese with reference to the other elements and to the σ phase. In the system used here, the formation of σ phase is associated approximately with the value of 7 electrons per atom, which is also the number of electrons per atom that manganese has. Manganese, it will be recalled, exists in three different crystal forms, none of which are the usual, simpler metallic forms; on the other hand, elements just to the left of manganese (in Table I) generally have lower coordination-number structures (body-centered cubic) than do elements to the right of manganese (face-centered cubic). The contribution to a complex crystal structure by the interalloying of elements from opposite sides of manganese is significant in that the σ phase is formed in binary systems including one element from each side of manganese, with manganese capable of participating with both sides.

It is indeed an interesting coincidence that manganese, with 7 electrons per atom, solidifies in complex crystal forms, and that when alloys of the transition elements are made which have seven 3*d*-4*s* electrons per atom, they, too, tend to assume a complex crystal form.

From the above, it might be deduced that the study of the σ phase might be advanced by a critical examination of the element manganese and how its structure is affected by small additions of elements which change the 3*d*-4*s* electrons per atom number. The role of atom size would also be of interest in σ formation.

Table I. Chart Showing Occurrence of the Sigma Phase in Binary Systems of Elements of the First Transition Group

	V	Cr	Mn	Fe	Co	Ni
V	—	—	σ	σ	σ	σ
Cr	—	—	σ	σ	σ	—
Mn	σ	σ	—	—	—	—
Fe	σ	σ	—	—	—	—
Co	σ	σ	—	—	—	—
Ni	σ	—	—	—	—	—
No. of electrons in 3d-4s levels per atom	5	6	7	8	9	10

Table II. Electrons/Atom Number for Various Sigma Phases

System	Boundaries for σ Phase in Atomic Pct at Temperatures Indicated	Electrons/Atom Ratio Corresponding to σ Phase Boundaries
V-Mn	24.3 pct V	6.5
V-Fe	37-57 pct V (700°C)	7.3 -6.9
V-Co	40-54.9 pct V	7.4 -6.8
V-Ni	55-65 pct V	7.2 -6.7
Cr-Mn	19-24 pct Cr (800°C)	6.84-6.78
Cr-Fe	43.5-49 pct Cr (600°C)	7.1 -7.0
Cr-Co	56.6-61 pct Cr	7.3 -7.2
Mo-Fe	47-50 pct Mo (1400°C)	7.1 -7.0
Mo-Co	59-61 pct Mo (1500°C)	7.23-7.17
Cr-Mo-Ni	37 Cr, 27 Mo, 36 Ni, 42 Cr, 32 Mo, 26 Ni (1250°C)	7.4 -7.0

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aime NEWS

Copper Symposium, IMD Lecture, Howe Memorial Lecture To Highlight Metals Branch Meeting Program

PROGRAM committees of the three Metals Branch divisions have evolved what promises to be one of the finest programs ever presented, for the 175th AIME General Meeting scheduled for the Statler Hotel, Los Angeles, February 16 to 19.

One of the outstanding features on the IMD agenda will be a symposium on the Mechanisms of Hardening of Alloys. Another highlight will be the IMD Lecture to be given by Kent Van Horn. Research in Progress session which has grown in significance in the past years, has become an integral part of the General Meeting program. Of interest to those attending the meeting will be two Titanium sessions. A Powder Metallurgy session also is scheduled.

EMD will present a symposium on Copper Smelting which will be divided into two sessions. Two other copper sessions are scheduled. EMD will also present two Lead and Zinc sessions. Two Aluminum-Magnesium sessions will be a feature part of the EMD program. There will be an EMD general luncheon.

Iron and Steel Div. will present the Howe Memorial Lecture. The lecturer will be Joseph Winlock of the Budd Co., Philadelphia. ISD will also present technical sessions on blast furnace and open hearth production.

ISD and EMD are scheduled to participate in a joint symposium on Recent Developments in the Constitution of Slags, Mattes and Molten Salt. A panel of experts with John Chipman as Chairman in a round table discussion is scheduled to discuss the symposium topic. EMD and MED will combine forces for a joint session on Educational Training.

Two sessions on Titanium and Uncommon Metals will be presented on Wednesday, February 18. In most of the technical sessions, ample time will be allowed for adequate discussion of papers discussed.

The Metals Branch dinner on Tuesday will be preceded by a cocktail party scheduled for 6 pm. The Metals Branch Council meeting is scheduled for Monday, February 16 at 9:30 am.

April 15, 1953 is the deadline for IMD papers to be presented at the Fall Meeting.

EXTRACTIVE METALLURGY DIV.

MONDAY, FEBRUARY 16

10:30 am

Copper

H. R. Hanley and T. H. Weldon, Associate Chairmen

Effect of Chloride on the Deposition of Copper in the Presence of Antimony and Arsenic: V. Hospadaruk and C. A. Winkler, McGill University, Montreal.

New Cornelia Copper Smelter of the Phelps Dodge Corp. at Ajo, Ariz.: J. W. Byrkit, Phelps Dodge Corp.

2:30 pm

Copper

W. G. Rouillard, Associate Chairman

Rehabilitation of Asarco's Perth Amboy Copper Refinery: G. H. Weis, D. A. Busch, H. K. Spaulding, G. B. Paulding, American Smelting & Refining Co., Perth Amboy, N. J.

Anode Casting at the Garfield Smelter: N. Alston, M. J. Winkel, American Smelting & Refining Co., Garfield, Utah.

Arc Furnace Equipment and its Operation at the Kennecott, Utah Refinery: H. A. Shaw, H. G. G. Whitton, Kennecott Copper Corp., Garfield, Utah.

2:30 pm

Lead and Zinc

L. P. Davidson and W. T. Isbell, Associate Chairmen

Strontium and Barium Salts for Control of Lead Content of Electrolytic Zinc: A. C. Jephson, G. D. Stendahl, American Smelting & Refining Co.

Sintering Zinc Concentrates on the Blackwell 12x168 Ft Machine: A. E. Lee, Jr., Blackwell Zinc Co.

New Jersey's Sterling Furnace: W. M. Peirce, New Jersey Zinc Co., New York.

2:30 pm

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Joint Symposium
See ISD Program

TUESDAY, FEBRUARY 17

9:00 am

Lead, Antimony, Zinc

W. T. Isbell and L. P. Davidson, Associate Chairmen

Chihuahua Slag Fuming Plant: V. R. MacDonald, American Smelting & Refining Co.

Vacuum Dezincing of Desilverized Lead Bullion: T. R. A. Davey, Broken Hill Smelters, Australia.
Development of a Continuous Updraught Sintering Process: W. R. Burrow, Courtesy of the Australian Institute of Mining & Metallurgy.
Improvements and Savings at the Laredo Antimony Smelter: J. C. Archibald, Jr., R. L. Kulpaca, National Lead Co.

9:00 am

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Joint Symposium
See ISD Program

9:00 to 11:30 am

Aluminum-Magnesium

Joint Session with MED

H. W. St. Clair and J. K. Richardson, Associate Chairmen

Production Expansion Program for the Aluminum Industry: S. W. Anderson, DPA, Washington, D. C.
Outlook for Production of Magnesium: F. J. Krenzke, Dow Chemical Co., Midland, Mich.
Economic Importance of Pegmatites: P. M. Tyler, Bethesda, Md.
Economies with Low Grade Coke and High Grade Iron Ore in Blast Furnace Operation: Walter C. Rueckel,

12:00 m

Physical Chemistry of Extractive Metallurgy Committee Luncheon Meeting

H. H. Kellogg, Chairman

2:00 pm

Aluminum-Magnesium

H. W. St. Clair and P. T. Stroup, Associate Chairmen

Experimental Production of Alumina from Anorthosite at Laramie, Wyo.: H. W. St. Clair, Bureau of Mines.
Use of Gas Engines in the Aluminum Industry: R. W. Bayerlein, the Nordberg Co.
Cost Factors in Utilization of Foreign Bauxite: A. Johnson, consultant.

2:00 pm

Educational Training

Joint Session with MBD

J. P. Speilman and Rush Spedden, Associate Chairmen

2:00 pm

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Joint Panel Discussion
See ISD Program

6:00 pm

Metals Branch Cocktail Party

7:00 pm

Metals Branch Annual Dinner

K. C. McCutcheon, Toastmaster

WEDNESDAY, FEBRUARY 18

9:00 am

Physical Chemistry of Extractive Metallurgy

O. C. Ralston and R. Schuhmann, Associate Chairmen

Vapor Pressure of Zinc in the Reduction of ZnS by Cu and Fe: A. W. Bethune, L. M. Pidgeon, University of Toronto, Toronto.
Vacuum Leaching of Aluminum from Aluminum-Silicon Alloys: M. J. Spendlove and H. S. Caldwell, Jr., U. S. Bureau of Mines.
Kinetics of the Reaction of Silver with Aqueous Cyanide and Oxygen: G. A. Deitz, J. Halpern, University of British Columbia.

9:00 am

Symposium on Copper Smelting

J. C. Kinnear, Jr. and L. L. McDaniel, Associate Chairmen

Hurley Furnace and Boiler Description and Design: E. A. Slover, Kennecott Copper Corp.
Reverberatory Furnace Practice at Noranda: J. N. Anderson, Noranda Mines, Ltd.
Modern Smelter Design: J. W. Byrkit, Phelps-Dodge Corp.

9:00 am

Titanium and Uncommon Metals

B. W. Gonser and N. C. Fick, Associate Chairmen

Production of Germanium: A. P. Thompson and J. R. Musgrave, Eagle Picher Co., Joplin, Mo.
Rare Earth Separation and Metallurgy at Ames: F. H. Spedding, Iowa State College, Ames, Iowa.
Ion Exchange in Wet Process Metallurgy: A. B. Mindler, C. F. Paulson, the Permutit Co.
High Vacuum Distillation of Beryllium: C. S. Pearsall, Massachusetts Institute of Technology, Cambridge, Mass.

12:00 m

General Luncheon and Business Meeting

T. D. Jones, Toastmaster

Speaker: Curtis L. Wilson, Dean, Missouri School of Mines and Metallurgy, Rolla, Mo.

2:00 pm

Symposium on Copper Smelting

L. L. McDaniel and J. C. Kinnear, Jr., Associate Chairmen

Reverberatory Furnace Combustion Practice: H. Foard, International Smelting & Refining Co.
Copper Converting Practice at Asarco Plants: F. W. Archibald, American Smelting & Refining Co.
Current Refractory Practice and Development in Copper Smelting: L. A. McGill, W. F. Rochow, Harbison-Walker Refractory Co.

2:00 pm

Titanium and Uncommon Metals

N. C. Fick and B. W. Gonser, Associate Chairmen

Thermodynamic Equilibrium for Reactions Involving Titanium Compounds: D. D. Wagman, T. R. Munson, W. H. Evans, National Bureau of Standards.
Thermodynamic Properties of Molybdenum Dioxide: N. A. Gokcen, Michigan College of Mining and Technology, Houghton, Mich.
System Ag_2O-B_2O_3: G. M. Willis, F. L. Hennessy, University of Melbourne, Australia.
Preparation of High Purity Iron at the National Bureau of Standards: G. Moore, National Bureau of Standards.

INSTITUTE OF METALS DIV.

SUNDAY, FEBRUARY 15

8:00 pm

Program Committee Meeting

MONDAY, FEBRUARY 16

9:00 am

Constitutional Diagrams

Vanadium-Oxygen Solid Solutions: A. U. Seybolt, H. T. Sumsion, General Electric Co., Schenectady, N. Y.
System Zirconium-Chromium: R. F. Domagala, D. J. McPherson, M. Hansen, Armour Research Foundation, Illinois Institute of Technology, Chicago.
System Zirconium-Copper: C. E. Lundin, Dow Chemical Co., Denver; D. J. McPherson, M. Hansen, Armour Research Foundation, Illinois Institute of Technology, Chicago.

9:00 am

Titanium Properties

Mechanical Properties of High-Purity Titanium-Aluminum Alloys: H. R. Ogden, R. I. Jaffee, D. J. Maykuth, Battelle Memorial Institute, Columbus, Ohio, W. L. Finlay, Rem-Cru Titanium, Inc., Midland, Pa.

Microstructure and Mechanical Properties of Iodide Titanium: F. C. Holden, H. R. Ogden, R. I. Jaffee, Battelle Memorial Institute, Columbus, Ohio.

Preferred Orientations in Iodide Titanium: Carl J. McHargue, J. P. Hammond, University of Kentucky, Lexington, Ky.

2:30 pm

Titanium—Constitutional Diagrams

Titanium-Carbon Phase Diagram: Irving Cadoff, J. P. Nielsen, New York University, New York.

Titanium-Nickel Phase Diagram: Harold Margolin, Elmars Ence, J. P. Nielsen, New York University, New York.

Titanium-Tungsten and Titanium-Tantalum Systems: D. J. Maykuth, H. R. Ogden, R. I. Jaffee, Battelle Memorial Institute, Columbus, Ohio.

Constitution of Titanium Rich Titanium-Chromium-Aluminum Alloys at 1800°F and 1400°F: J. L. Taylor, Pol Duwez, California Institute of Technology, Pasadena, Calif.

Titanium-Manganese System: D. J. Maykuth, H. R. Ogden, R. I. Jaffee, Battelle Memorial Institute, Columbus, Ohio.

Habit Plane in Quenched Ti-Mn Alloys: Y. C. Liu, Harold Margolin, New York University, New York.

2:30 pm

Plastic Deformation

Effect of Tensile Strain at Low Temperatures on Deformation Twinning in Ingot Iron: Glenn W. Geil, N. L. Carwile, National Bureau of Standards, Washington, D. C.

Directional Properties of 2S Aluminum: K. T. Aust, F. R. Morral, Kaiser Aluminum & Chemical Corp., Spokane, Wash.

Bend Plane Phenomena in the Deformation of Zinc Monocrystals: J. J. Gilman, General Electric Co., Schenectady, N. Y., T. A. Read, Columbia University, New York.

A Qualitative Crystallographic Study of the Recrystallization of a Critically Oriented Cold Rolled Copper Single Crystal: Y. C. Liu, New York University, New York, W. R. Hibbard, Jr., General Electric Co., Schenectady, N. Y.

Cold Rolling and Annealing Textures of Molybdenum Single Crystals: N. K. Chen, R. Maddin, Johns-Hopkins University, Baltimore, Md.

TUESDAY, FEBRUARY 17

9:00 am

Metallography

Statistical Grain Structure Studies: Plane Distribution Curves of Regular Polyhedrons: F. C. Hull, W. J. Houk, Westinghouse Electric Corp., Pittsburgh.

12:15 pm

Executive Committee Luncheon Meeting

2:00 pm

Research in Progress

(Abstracts of Papers to be presented at this session will appear in the February issue of JOURNAL OF METALS)

6:00 pm

Metals Branch Cocktail Party

7:00 pm

Metals Branch Annual Dinner

K. C. McCutcheon, Toastmaster

WEDNESDAY, FEBRUARY 18

9:00 to 10:30 am

Zr, Ti Plastic Deformation

Kinetics of Thermal Reorientations in Cold-Rolled Zirconium: R. K. McGeary, B. Lustman, Westinghouse Electric Corp., Pittsburgh.

Mechanism of Plastic Flow in Titanium: Determination of Slip and Twinning Elements: F. D. Rosi, Sylvania Electric Products, Inc., Bayside, L. I., C. A. Dube, Laval University, Quebec, B. H. Alexander, Sylvania Electric Products, Inc., Boston.

Creep Properties of Commercially Pure Titanium: W. R. Kiessel, Ford Scientific Laboratory, Dearborn, M. J. Sinnott, University of Michigan, Ann Arbor.

10:45 am

Annual Business Meeting

W. A. Dean, Chairman

11:00 am

Annual Lecture, IMD

M. L. Haider and W. A. Dean, Associate Chairmen

Residual Stresses Introduced During Metal Fabrication: Kent R. Van Horn, Aluminum Co. of America, New Kensington, Pa.

2:00 pm

Symposium on Mechanisms of Hardening of Alloys

A series of three lectures on the mechanism by which alloys can be hardened. The lecturers will present a critical review of the present status of knowledge and express their own points of view. Time will be available for discussion from the floor.

Martensite Reaction: A. R. Troiano, Case Institute of Technology, Pittsburgh.

Precipitation Hardening: W. L. Fink, Aluminum Co. of America, New Kensington, Pa.

Heat Treatment of Titanium Alloys: Pol Duwez, California Institute of Technology, Pasadena.

THURSDAY, FEBRUARY 19

9:00 am

Gas Metal Reactions and Powder Metallurgy

Observation on Scaling of Iron: W. J. Wrazej, Dept. of Mines and Technical Surveys, Ottawa.

Density and Hydrogen Occlusion of Some Ferrous Metals: J. H. Keeler, General Electric Co., Schenectady, N. Y., H. M. Davis, Pennsylvania State College, State College, Pa.

9:00 am

Creep of Metals

Nature of the Creep Curve: T. H. Hazlett, E. R. Parker, University of California, Berkeley.

Grain Boundary Sliding and Migration and Intercrystalline Failure under Creep Conditions: H. C. Chang, N. J. Grant, Massachusetts Institute of Technology, Cambridge.

Some Observations on Correlations Between the Creep Behavior and the Resulting Structures in Alpha Solid Solutions: O. D. Sherby, J. E. Dorn, University of California, Berkeley.

12:15 pm

Powder Metallurgy Committee Luncheon

2:00 pm

Transformation and Thermodynamics

Occurrence and Characteristics of Chi Phase: P. K. Koh, Allegheny Ludlum Steel Corp., Brackenridge, Pa.

Absolute Rate Theory Applied to Rate of Growth of Pearlite: J. H. Frye, Jr., Oak Ridge National Laboratory, Oak Ridge, Tenn., E. E. Stansbury, University of Tennessee, Knoxville, Tenn., D. L. McElroy, University of Alabama, University, Ala.

Determination of the Absolute Grain Boundary Energy of Gold at 1300°K: F. H. Buttner, Minerals & Metals Advisory Board, Washington, D. C., U. Udin, J. Wulff, Massachusetts Institute of Technology.

2:00 pm

Symposium on the Structural Use of Powder Metallurgical Parts

George D. Cremer, Chairman

1. Parts from Elemental Powders
2. Parts from Alloy Powders
3. The Contribution of Infiltration Techniques
4. The Solution to High Temperature Problems Offered by Cermets
5. The Promise in Hot Pressing and Working

IRON AND STEEL DIV.

MONDAY, FEBRUARY 16

9:00 am

Blast Furnace

John Saussaman and D. T. Rogers, Associate Chairmen

Taconite Development in Minnesota. Color sound motion picture.

Chemical Reactions of Coke in the Blast Furnace: J. F. Peters, Inland Steel Co., Chicago.

Physical Conditions in the Combustion and Smelting Zones of the Blast Furnace: J. F. Elliott, Inland Steel Co., E. Chicago, Ind., R. A. Buchanan, J. B. Wagstaff, U. S. Steel Co., Kearny, N. J.

Aluminum-Oxygen Equilibrium in Liquid Iron: N. A. Gokcen, Michigan College of Mining and Technology, Houghton, Mich., John Chipman, Massachusetts Institute of Technology, Cambridge.

Reduction in Silicon from Blast Furnace Type Slag: J. C. Fulton, John Chipman, Massachusetts Institute of Technology, Cambridge.

2:30 pm

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Joint Symposium with EMD

H. H. Kellogg and M. Tenenbaum, Associate Chairmen

Chemistry of Iron Silicate Slags: R. Schuhmann, Jr., Massachusetts Institute of Technology, Cambridge.

Oxygen Activity in Iron Oxide Slags: H. Larson, J. Chipman, Massachusetts Institute of Technology, Cambridge.

Electrochemistry of the Iron Oxide Silica System: Nature of the Conductivity: M. Simnad, G. Derge, Carnegie Institute of Technology, Pittsburgh.

Manganese and Phosphorus Oxidation During and Just After the Flush Period in Basic Open Hearth Practice: J. F. Elliott, F. W. Luerssen, Inland Steel Co., E. Chicago, Ind.

Observations on the Properties and Behavior of Acid Open Hearth Slags in Relation to Operating Variables: G. R. Fitterer, University of Pittsburgh, Pittsburgh.

Reducing Period in Stainless Steel Melting: H. P. Rassbach, Electro Metallurgical Co., Div. of UCC Corp., Chicago.

Solutions of Metals in Fused Salts: D. D. Cubicciotti, North American Aviation,

TUESDAY, FEBRUARY 17

9:00 am

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Joint Symposium with EMD

G. Derge and R. Schuhmann, Associate Chairmen

Electrical Properties and Constitution of Molten Mattes: Iron-Copper System: G. M. Pound, G. Derge, Carnegie Institute of Technology, Pittsburgh.

Diffusion of Calcium in Blast Furnace Type Slags: H. Towers, John Chipman, Massachusetts Institute of Technology, Cambridge.

Silicon and Manganese Deoxidation Under Unsaturated Slags: N. Christensen, John Chipman, T. Hartley-Smith, Massachusetts Institute of Technology, Cambridge.

10:45 am

Annual Business Meeting

K. C. McCutcheon, Chairman

11:00 am

Howe Memorial Lecture

Andrew Fletcher and K. C. McCutcheon, Associate Chairmen

Influence of the Rate of Deformation on the Tensile Properties of Some Plain Carbon Sheet Steels: Joseph Winlock, Budd Co., Philadelphia, Pa.

2:00 pm

Recent Developments in the Constitution of Slags, Mattes and Molten Salts

Round Table Discussion

Joint Session with EMD

John Chipman, Chairman

Panel of Experts

6:00 pm

Metals Branch Cocktail Party

7:00 pm

Metals Branch Annual Dinner

K. C. McCutcheon, Toastmaster

WEDNESDAY, FEBRUARY 18

9:00 to 10:30 am

Gases in Steel

G. Derge and C. R. Taylor, Associate Chairmen

Attainment of Equilibrium in Gas Metal Reactions: N. A. Gokcen, Michigan College of Mining and Technology, Houghton, Mich.

Thermal Conductivity Method for Analysis of Hydrogen in Steel: B. M. Shields, U. S. Steel Co., Duquesne, Pa., John Chipman, N. J. Grant, Massachusetts Institute of Technology, Cambridge.

2:00 pm

STEEL PRODUCTION

C. M. Squarcy and H. P. Rassbach, Associate Chairmen

Use of Oxygen in the Bessemer Converter: W. T. Rogers, L. T. Sanchez, U. S. Steel Co., Lorain, Ohio.

Activity of Sulphur in Liquid Iron and Steel: C. W. Sherman, John Chipman, Massachusetts Institute of Technology, Cambridge.

An Evaluation of the pH and Conductivity Methods of Slag Control: P. D. S. St. Pierre, Dept. of Mines and Technical Surveys, Ottawa.

Accelerated Solidification in Ingots: Its Influence in Ingot Soundness: E. Marburg, U. S. Steel Co., Pittsburgh.

To Company Executives: How Many of Your Staff Are Going To The 1953 Annual Meeting?

By Edward H. Robie,
Secretary, AIME

In our recent visits to some fifteen Local Sections we have tried to impress on members the value of attendance at Institute meetings, specifically the Annual Meeting this year in Los Angeles where some 2500 professional men will forgather. In many instances, particularly with the younger men, the response has been a rather wistful smile with the remark that "I certainly would like to be there." They want like the devil to go, and feel they would get a great deal out of it, but they are pretty sure they will not be allowed to go. They all work for some one else, and it is the top brass that all too often takes such trips.

We recall the first job that we ever had in the mining industry. Not long after we were employed—three years to be exact—we were sent, not to an AIME meeting, but on a field trip of several weeks to investigate operating practice of other companies. The aim was the same as attendance at a meeting, but the trip was more costly in time and money. Its purpose was primarily to learn things that would save our employer money, and to make us a more valuable employee to them. Incidentally, our wife was sent along with us, and it served as our honeymoon. We feel that our later report to the company was worth what it cost, but we may be prejudiced. Certainly, the company thereby secured an enthusiasm in our work, and a staunch friend for years to come.

Employers, we believe, should give serious thought to which members of their staffs should attend the Los Angeles meeting. Other factors being reasonably equal, AIME members should certainly be preferred to nonmembers, else the members will naturally believe that their membership counts nothing. The trip should be looked on as an investment, and those selected to go should be those that have shown by their work that they are most likely to profit by the contact with others in their line of work. They should be required to make some kind of a report of what they have learned, the people they have met, and the ideas they have developed. But not only is the return to the employer the increased knowledge attained by the employee, but the great improvement likely in his morale. He will be a better friend of the company, more satisfied with his job.

The less liberal employer will merely give permission to attend the meeting, without paying expenses. Even this would be deeply appreciated by many young men, especially those not too distant from Los Angeles. No operation is likely to suffer much because of anybody's absence for a week. Operations continue even when one is ill, or on vacation. A more liberal attitude is to pay expenses as well as to accord permission. And if an employer wants to make a still greater investment, with higher dividends, he will send both the man and his wife. More and more companies are doing this.

It is just as important to keep the human tool sharp and bright as it is the machine.

Hollywood Bowl, where music under the starry California night is one of the many great attractions California has to offer. The varied highlights of Los Angeles will be at the disposal of Annual Meeting registrants from the centrally located Statler Hotel.

Seated at luncheon are the people who are helping to make the 1953 Annual Meeting a successful reality. Clockwise, beginning at head of table: Mrs. R. H. Jahns, Chairman, WAIMME Registration; Newell Appleton, Assistant Secretary, AIME; Ian Campbell, Chairman, Banquet; W. H. Geis, Chairman, Reception; A. C. Rubel, Chairman, Welcoming Luncheon; J. H. Pemberton, Chairman, Hotel; Henry Mulryan, Chairman, Field Trips; Blair W. Stewart, Secretary; C. E. Totten, Vice Chairman, Cocktail Party; James D. Hughes, Chairman, Finance; Evan White, Assistant Secretary; R. E. O'Brien, Field Secretary, AIME; John R. West, Vice Chairman, Publicity; Thomas F. Edson, Chairman, Informal Dance; Laurence Chasteen, Chairman, Technical Sessions Arrangements; E. A. Arthur, Vice Chairman, Field Trips; Nicholas A. D'Arcy, Jr., Vice Chairman, Annual Meeting; Frederic Boericke, Vice Chairman, Annual Meeting, and Chairman, Registration; Harold Clark, Chairman, WAIMME Liaison; Mrs. Harold Clark, General Chairman, WAIMME Annual Meeting; and Henry T. Mudd, General Chairman, 1953 Annual Meeting.

Annual Meeting News and Notes

John H. Chesters Awarded Hunt Medal

K. C. McCutcheon, Chairman of the Robert W. Hunt Medal and Prize Committee, announced that the committee had selected J. H. Chesters, assistant director of research, United Steel Companies, Ltd., of Sheffield, England, to receive the award in 1953. The award is being presented to Mr. Chesters for his paper entitled, *Flow Patterns in Open-Hearth Furnaces*, published in the 1951 Open Hearth Proceedings. A silver medal will be awarded to Mr. Chesters at the Annual Banquet in February, to which he will be invited as a guest of honor of the Institute.

Mathewson Gold Medal Winners Named

The following selections as winners of the Mathewson Gold Medal for 1953 were announced to the Board, as presented by M. B. Bever, Chairman of the Mathewson Gold Medal Committee: Paul A. Beck, Hsun Hu, and P. R. Sperry. The award-winning series of closely related papers, published in JOURNAL OF METALS Transactions, are: *Annealing Textures in Rolled Face-Centered Cubic Metals*, by Paul A. Beck and Hsun Hu; *Rolling Textures in Face-Centered Cubic Metals*, by Hsun Hu, Philip R. Sperry, and Paul A. Beck; and *Recrystallization Texture and Coarsening Texture in High Purity Aluminum*, by Paul A. Beck and Hsun Hu.

Each will receive the Mathewson Gold Medal during the Annual Meeting in Los Angeles in February and will be guests of honor at the Annual Banquet.

Student Activities To Be Held At Annual Meeting

Student Associate members of the AIME can attend the 175th General Meeting without payment of the registration fee. Nonmember students will be required to pay $1.

All registered students will be given badges admitting them to technical sessions. Students will also be presented with a packet of technical papers without charge.

Open to students without charge will be the dinner and panel discussion *Modern Mineral Engineering Curricula*, sponsored by Mineral Industry Education Div. A charge of $5.00 will be made to students desiring to attend the Welcoming Luncheon. Admission to the entertainment portion of the Stag Dinner will be without charge to students. AIME members are being asked to act as sponsors for students desiring to attend the dinner as well.

A special student session, *Opportunities in Mineral Engineering*, has been arranged, with six prominent AIME members scheduled to address the group.

C. M. Squarcy To Receive Johnson Award

Kurt Neustaetter, Chairman of the J. E. Johnson, Jr., Award Committee, announced to the Board that the committee had selected Charles M. Squarcy, assistant superintendent blast furnaces, Inland Steel Co., East Chicago, Ind., to receive the J. E. Johnson, Jr., Award for 1953. The citation offered to accompany the award reads as follows: "for his ability as a blast-furnace operator, for his contributions to blast-furnace literature, and for his work in building this professional organization." Mr. Squarcy will be invited to be a guest of honor of the Institute at the Annual Banquet.

Annual Meeting Field Trips Planned

Several field trips are being contemplated for Feb. 20 and 21 as adjuncts to the AIME Annual Meeting. Those expecting to attend the Los Angeles gathering have been asked to indicate their preferences.

Among the trips thus far contemplated are: Riverside Cement Co. (Crestmore mine), Kaiser Steel Co. (Fontana plant and Eagle Mountain mine), mines, oil fields and refineries, petroleum research laboratories, cement companies, steel and metal fabricating plants, automobile and aircraft plants, rock product companies, aerial trips over Los Angeles and Catalina Island, Palomar Observatory, and others.

Special Tour Planned

AIME members from the east are invited to travel to Los Angeles via the AIME special train from Chicago. This train will leave Chicago at 11:55 am (Central Time) on Friday, Feb. 13, 1953 and stop at Salt Lake City on Saturday. There will be a 3 hr tour of the Utah open cut copper mines in Bingham Canyon, and a visit to the unique western mining town of Bingham.

The train will leave Salt Lake City at 9:00 pm (Mountain Time) and arrive at Las Vegas on Sunday. There will be a bus tour to Boulder City and Hoover Dam, and a sightseeing tour at the casinos of Las Vegas. The train will be boarded at 9:00 pm (Pacific Time) Sunday, and the last stop will be Los Angeles, 7:30 am Monday. Interested persons should make reservations now by writing J. J. Cummins, Traveling Passenger agent, Union Pacific R.R., 1 S. LaSalle St., Chicago 3.

Bessemer Men to Meet

Russell C. Buehl, chief, ferrous pyrometallurgy section, U. S. Bureau of Mines will present the discussion *Air Oxidation of High Phosphorous Spiegeleisen in a Basic Converter* at the January 14 meeting of the Bessemer Committee, Duquesne Club, Pittsburgh. Fred Skeates, general superintendent, Link-Belt Co., Chicago is scheduled to present *Side Blowing Bessemer Foundry Practice*. In addition the following subjects on acid bessemer ingot production will be discussed: Mixers, converter operation, iron and steel quality, rolling mill practice, and new developments at home and abroad.

Metals Branch Deficit Discussed at Meeting

More time was given by the Board at its Nov. 19 meeting to the matter of financing the Metals Branch deficits than to any other subject. Walter A. Dean presented a report of the Committee to Study Sustaining Memberships for the Metals Branch, of which he was Chairman. A total of 1000 pages of Metals Branch Transactions in 1953 was thought desirable, 750 pages for IMD, and the balance for EMD and ISD. A part of this material would be issued as supplements to the JOURNAL OF METALS. If this amount of Transactions material were published, a deficit of $20,000 to $25,000 could be expected in the Metals Branch in 1953. To meet this deficit, contributions of from $200 to $500 would be solicited from industrial concerns, institutional groups, and individuals interested in supporting publication of this type of material. After long discussion the directors voted 9 to 7 against adoption of the proposed plan for contributions at this time. Instead, the Board took the position that for the coming year the deficit to be incurred by the proposed publishing plan should be met out of the general income of the Institute, as it has in the past. The matter is to be reconsidered if it later appears advisable to do so.

Discuss Latin American Steel Production Problems

Problems relating to the expansion of steel production in Latin America and new processes that might be applicable to the raw materials of this area were discussed at the Primera Conferencia Siderurgica Latina Americana, which was held in Bogotá, Colombia, from Oct. 13 to 31, 1952. Outstanding metallurgists from North and South America and Western Europe were invited to participate. In addition to 66 specialists from 7 Latin American countries, other countries represented were: 15

from United States; 13 from France; 6 from West Germany; 2 each from Norway, Switzerland, and United Kingdom; 1 each from Belgium, Canada, Denmark, and Sweden.

Excellent papers and discussions were given on the following subjects: (1) Various methods for cleaning coals. (2) Preparation of coke from a variety of coals, especially those of Latin America. (3) The charcoal blast furnace. (4) Methods for reducing ores otherwise than in the blast furnace. (5) Comparison of open hearth, converter, and duplex processes for steelmaking. (6) Production of steel shapes by continuous casting, extrusion, and reduction of preformed ore. (7) Application of steels made by different processes. (8) Influence of local factors on Latin American steelmaking practice.

Supplement To Be Issued

The February issue of JOURNAL OF METALS will be published in two sections to expedite and facilitate the handling of the large number of Transactions papers prepared and submitted in time for presentation at the AIME Annual Meeting in February. Section 2 will contain exclusively the overflow IMD Transaction papers from Section 1. A two-section journal will probably be issued three or four times a year to coincide with the surge of papers created by the various meetings of the Metals Branch of the Institute.

To Invest AIME Funds in Common Stock

At the recent Board meeting, consideration was given investing an increased portion of AIME funds in common stocks rather than bonds or preferred stocks. Permission was voted to allow the Investment Committee to invest up to 50 pct of Institute funds in common stocks except where special conditions or limitations exist, as in certain specific Funds. The instruction was permissive and not directive.

Petroleum Branch To Issue Cumulative Index

A cumulative index of petroleum papers published by the AIME to date is to be issued by the Petroleum Branch. Its cost will be underwritten by the Branch's Special Projects Fund and it is expected that sales to members will cover at least half the cost. Papers in the Transactions volumes will be indexed, and also other important material in *Petroleum Technology* and the JOURNAL OF PETROLEUM TECHNOLOGY. The last all-Institute cumulative index covered material published through 1936.

AIME Branches Meet at Seventh Annual Off-the-Record Meeting Held in Pittsburgh

The seventh annual Off-the-Record meeting of the Pittsburgh Section of AIME and the National Open Hearth Committee, held in Pittsburgh, Pa., Nov. 7, 1952, was an outstanding success. Registration was 670, a record for these meetings. Quality of the technical sessions was considered the best in the history of the gathering.

All day technical programs were presented by the Coal, Petroleum and Institute of Metals Div. of the Pittsburgh Section of AIME. The Pittsburgh Section of the National Open Hearth Committee presented three technical programs under the subjects of *operating, metallurgy* and *refractories and masonry*. A luncheon was held at the hotel. Activities for the day were concluded with a cocktail party and dinner also held in the hotel. Principal dinner speaker was Lew Worsham, Oakmont Country Club professional, who gave a demonstrated lecture on golfing. Guests at the speakers table included E. H. Robie, and E. O. Kirkendall, Secretary and Assistant Secretary of AIME and M. L. Haider, President of AIME.

The Coal Div. program considered three major problems of the industry—mining, preparation, and utilization. Of particular interest was a paper entitled *Review of Factors Affecting Coal Preparation Plants* by O. R. Lyons, R. L. Llewellyn, and J. A. Younkins. Another paper that attracted attention was *Modification and Use of the Movable Wall Experimental Coke Oven* by P. E. Jordan. A three dimensional movie on coal sludge recovery, produced by the Coal & Mineral Washer Corp. and presented by R. A. Stiveson, held novelty appeal as well as being informative. The program concluded with a paper *Trackless Mining Thin Seams* presented by R. A. Ison.

Five papers were presented at the Petroleum Div. session. Seven papers and three movies were presented at the Institute of Metals session. In one of the two papers on aluminum, J. A. Nock, Jr. discussed the use of 14S, 24S, 75S and XA78S alloys in aircraft and the welding by inert gas shielded arc processes of Al-Mg alloys. In the second paper, O. M. Mader described utilization in the new Alcoa office building, downtown Pittsburgh, of many innovations, including aluminum outside walls, pneumatically sealed windows, overhead heating and cooling systems, and general lightweight construction. J. H. Bechtold discussed the recrystallization behavior of molybdenum and pointed out the wide variation in mechanical properties that can be expected with variations in grain size. H. T. Clark in another interesting paper described and classified titanium base alloys. Included in the classification were the desirable and undesirable characteristics of each alloy. R. H. English described the characteristics of a new alloy, NA22H, designed for service at extra high temperatures. *885°F Embrittlement in Stainless Steels* was the title of Dr. A. J. Lena's paper.

Fourteen subjects were discussed in the three sessions of the National Open Hearth Committee. Following the usual custom at these meetings, prepared discussions were often presented by more than one speaker and opportunity for informal discussion from the floor was offered. In the operating session two of the most interesting subjects were *High Percentage of Hot Metal and Charged Ores—Effect on Yield and Production* and *Mold Life Comparisons*. Discussions by J. Turner on the first, and by D. J. Taylor on the second paper prompted a great deal of comment from the floor. After his presentation on *Autopour*, R. P. Carpenter was asked numerous questions about type, desirable pressures, and increases in pour rates that could be expected. L. H. Veiock described an immersion thermocouple used by the Heppenstall Co. to determine its open hearth bath temperatures.

The metallurgy session opened with a discussion of the application of the direct reading spectrograph to open hearth control. Participants agreed this equipment has demonstrated its value for the rapid and economical determination of residuals. A discussion of aluminum killed steels for deep drawing covered various methods of making aluminum additions. The search continues for a good mold coating to replace generally used tar. The requirements for a satisfactory mold coating were discussed and reviewed for a variety of materials, proprietary and otherwise. A discussion of hot scarfing machines for slabs intended for sheet created much interest. It seemed evident that in spite of the high cost of mechanical scarfers of this size, their installation may in some cases be justified because of the substantial reduction in the amount of steel that must be hand-scarfed.

The presentations in the refractories and masonry session included *Open Hearth Bottoms, Expansion Allowances in Open Hearth Constructions* (including both silica and basic brick), and *Pictorial Record of Construction of Modern Open Hearth Plant*. Presentations were given by suppliers and users of refractories.

At the Annual Dinner, attended by most of the Conference registrants, T. F. Dorsey served as Toastmaster, and W. P. Dudley was Speaker. Past Chairmen of the Executive Committee received awards for their service.

Electric Furnace Steel Conference Doubles Attendance In Ten Years — Past Chairmen Honored

Wide Variety of Subjects Discussed at Formal and Informal Sessions

The Tenth Annual Electric Furnace Steel Conference was celebrated in grand style, with attendance almost exactly double that of the first conference in 1943. Some 829 interested persons joined in technical sessions designed to be of interest to the greatest number of steel men. Although it would not be correct to extrapolate attendance figures and predict a doubling of registration every 10 years, it is perfectly safe to state that the Electric Furnace Conference has definitely come into its own.

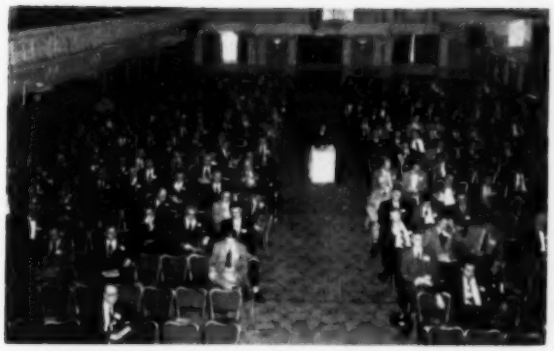
The purpose of these meetings is to provide a means of interchange of developments and ideas for the good of the individuals and industry. That this ideal has been reached can be seen by the number of impromptu meetings held after and between scheduled sessions, with operating personnel from different companies comparing notes and bringing up various problems.

The formal sessions opened with remarks by W. M. Farnsworth, Conference Chairman, and R. H. Frank, Executive Committee Chairman. At the general session that followed, much interest was evidenced in the paper by Rudolph Smith, CF&I, on *Human Relations in the Furnace Shop*. Many companies have extensive programs underway in this field, and have found that these programs pay off in many ways. It was pointed out that there is no formula for a good human relations program, but each plant must develop its own system and support it. Statistical analyses are becoming less of a rarity in the steel industry. C. R. Taylor, Armco Steel Corp., discussed the advantages and reliability of multiple correlations as against simple correlations. Simple correlations can lead to misleading results. D. J. Girardi presented a resumé on use of oxygen in electric furnaces.

Ray Frank, Executive Committee Chairman presents perpetual registration and certificate to Tom McLoughlin, Past Chairman. T. F. Dorsey and W. M. Farnsworth, Conference Committee Chairman, look on.

The fume control session was considered by many of those attending to be one of the most interesting and controversial sessions held. As more cities adopt regulations to control air pollution, the pressure on furnace shops to control fumes increases. There is not as yet any concrete answer to the problem, but much work is in progress on all possible methods. This subject is one that involves the community and top management as well as operating personnel, and will become more intense as time goes on.

On Friday, simultaneous sessions were held; castings technical sessions and ingot technical sessions. On Saturday, an educational session, Procedure in Making a Heat of Stainless Steel was held. These annual educational sessions are planned to help melters, helpers, observers, students, and young metallurgists,



In addition to a general session, simultaneous casting and ingot sessions were held to appeal to the widest audience. This is a view of the opening session of the Conference.

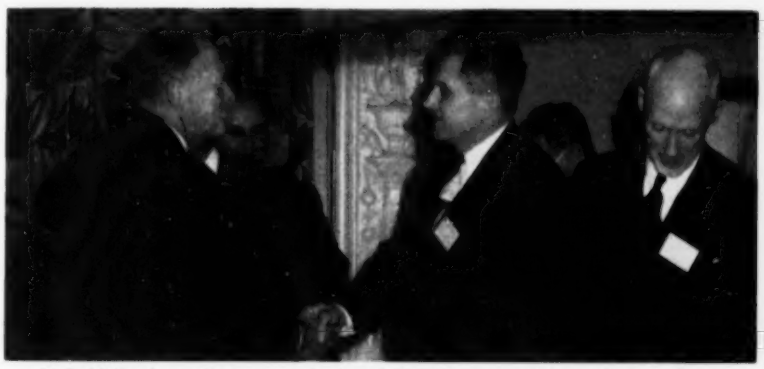
As important as the formal sessions are the informal sessions. In addition to renewing old acquaintances, many operating problems are threshed out.

and to serve as a refresher course to regular registrants.

At the Annual Dinner on Thursday evening, T. J. McLoughlin, Past Chairman of the Executive Committee received his perpetual registration to the conferences and a certificate. Ray Frank revealed a well kept secret with the awarding of 400-day clocks, each suitably engraved to H. W. McQuaid, C. W. Briggs, W. J. Reagan, J. B. Caine, N. I. Stotz, J. A. Bowers, and T. J. McLoughlin, Past Chairmen of the Electric Furnace Steel Executive Committee. Clocks were also awarded to R. L. Baldwin, one of the original organizers, and Frank Sisco, first Secretary, for their pioneer work in the early days of the committee.

T. F. Dorsey, president of Pittsburgh Steel Foundry Corp. served as toastmaster, and introduced those seated at the head table. This year, the foundry industry was well represented, filling most of the seats of honor. Mr. Dorsey then introduced K. C. McKutcheon, Chairman of the Iron & Steel Div., who spoke as official representative of AIME, and welcomed all to the meeting. The toastmaster then presented the speaker of the evening, W. P. Dudley, vice-president in charge of operations, Ohio Steel Foundry Co. Mr. Dudley's theme was that all matters are governed by law, whether spiritual, moral, economic, or other. His talk, which alternated the light and the serious captured the attention of the audience, and was well received.

In the December Electric Furnace Conference Issue of JOURNAL OF METALS, articles by Harry W. McQuaid and T. J. McLoughlin on the history and evaluation of the conferences, appeared side by side. Although no credit can be taken for the coincidence, the Editor learned at the conference that Harry McQuaid and Tom McLoughlin sat side by side in class at Stevens Institute of Technology. Another classmate was Jerome Strauss, also in attendance at the conference.



K. C. McKutcheon, Chairman of the Iron & Steel Div. congratulates W. P. Dudley, Ohio Steel Foundry Co., on his fine dinner talk.

Harry McQuaid, Norman Stotz, W. J. Reagan, and Jim Bowers, stand behind Tom McLoughlin and one of the 400-day clocks awarded them for their service to the Conference as Past Chairmen.

Officers for the Eleventh Annual Electric Furnace Steel Conference, to be held at the Netherlands Plaza Hotel, Cincinnati, Dec. 3 to 5, 1953, are W. M. Farnsworth, Chairman of the Executive Committee; F. O. Lemmon, Chairman of the Conference Committee; and H. F. Walther, Vice-Chairman of the Conference Committee.

Connecticut Section Holds Successful Meeting

A novel departure from the usual procedure was experienced when the Connecticut Local Section devoted one of its regular meetings to steel, and journeyed to Hartford for the meeting. Through the courtesy of Walter Jacob, chairman of the board, Hartford Electric Steel Co., a plant visit was arranged through that company. Hartford Electric specializes in alloy castings of most ferrous alloys. All plant officials were on hand to welcome the group, and helped guide the tour.

The meeting, held at the Hartford Club, was preceded by a cocktail party provided by Mr. Jacob. Leon Thelin, Section Chairman, gave the group a brief history of AIME and the founding of the Connecticut Section. Bill Mounce was Chairman for the session. Speaker for the evening was Clayton Berry of Hartford Electric Steel Corp., who spoke on *The Use of Rare Earth Additions to Improve the Properties of Steel Castings.* Mr. Berry, no newcomer to the rare earth field, has been studying the effects of the rare earths for the past 14 years. His talk dealt not only with the use of the rare earths, but also with their chemistry, and some of the mechanisms accounting for their beneficial effects. One of the most interesting items was that he was able to attain successful use of mischmetal in acid electric practice, the trick being to take precautions against the rare earth addition being mixed with the slag. The greatest benefit derived from rare earth additions was where specifications call for high impact strength at very low temperatures. He took pains to point out that rare earths were not a cureall, and their additional cost had to be weighed against the benefit derived, if the superior properties were necessary to the design, and the customer would pay.

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Journal of Metals To Give Conference Award

Blast Furnace, Coke Oven and Raw Materials Conference papers submitted and accepted by the Conference Program Committee by January 15 will be eligible for the new annual JOURNAL OF METALS Award. The award, a $100 check and a suitably inscribed certificate will go to the author or authors of the best paper presented at the conference. Presentation to the winner will be made at the Annual Fellowship Dinner. The first JOURNAL OF METALS Award will be given in April 1953.

Chairman of the Blast Furnace, Coke Oven and Raw Materials Committee will select five members of the AIME to sit on the award committee. Members of the award committee must have served at least six months as an officer, member of the executive board, or member of the conference committee of the Blast Furnace, Coke Oven, and Raw Materials Committee.

Papers will be weighed on the basis of 20 points for originality, 40 points for practical value, 25 points for scientific value, and 15 points for presentation. In cases of joint authorship separate certificates will be presented, and the $100 will be divided.

January 15 has been set as the deadline for acceptance during each year of the conference. No eligibility limitations have been set because of age, sex, prior receipt of the award, or nonmembership in the AIME.

To Release 1952 Transactions Volumes

AIME Transactions volumes 193, 194, and 195, which are respectively the Mining, Metals and Petroleum volumes will be released on Mar. 15, 1953. The volumes correspond to the Transactions sections of the three branch publications that were published in 1952. The price for these books is $4.90 to Members and $7.00 to nonmembers, however, if ordered and paid for with the dues, the price is only $3.50 for Members.

No Change In Institute Name Planned

All discussions regarding any suggested change in the name of the Institute, have indicated that there is no particular desire for any change. No enthusiasm has arisen for the name "American Institute of Mineral Engineers." "American Institute of Mining, Metallurgical and Petroleum Engineers" is too unwieldy. "American Institute of Miscellaneous Enginners" is only good for a laugh. One suggestion is perhaps worthy of consideration: On all letterheads, carry the names of the ten Divisions of the Institute.

New Local Sections Formed in Southwest

A new Local Section of the AIME, to be known as the West Central Texas Section, has been formed. Headquarters will be in Abilene. Its area embraces the following counties, which have been released by the Permian Basin and North Texas Sections: Scurry, Mitchell, Stonewall, Fisher, Nolan, Coke, Haskell, Jones, Taylor, Runnels, Shackelford, Callahan, Coleman, McCullough, Stephens, Eastland, Comanche, Brown, Mills, San Saba, Hamilton, Lampasas, and Coryell.

An application and bylaws for the formation of the Lou-Ark Local Section were approved at the recent meeting of the Board of Directors. The Section will include all that area in the states of Arkansas and Louisiana previously included in the East Texas Section which has signified its willingness to release this area. The area is bounded on the west by the western boundaries of Louisiana and Arkansas, on the north by an east-west line passing through Hot Springs Station across Arkansas from Oklahoma to the Mississippi River, on the east by the Mississippi River, on the south by a straight line beginning at the Sabine River, passing through Torras and Fullerton, La., and terminating at the Mississippi River. This Section will be among those administered by the Petroleum Branch.

Approval of the transformation of the South Plains Subsection of the Permian Basin Section to the South Plains Local Section has been given by the Board of Directors. The Permian Basin Section approved the release of this territory. The new Section will include the following counties of Texas: Bailey, Yoakum, Lubbock, Crosby, Dawson, Dickens, Floyd, Cochran, Cottle, Hale, Hockley, Kent, King, Gaines, Garza, Lynn, Motley, and Terry. This Section will also be administered by the Petroleum Branch.

Personals

J. P. Dempsey formerly with the Potash Co. of America, Dumas, Texas is now with the Blue Diamond Corp., Blue Diamond, Nev.

Eric C. Bell has joined Factory Mutual Engineering Div., Toronto as engineer-inspector. He had been with the Ontario Research Foundation.

Julian Miles Avery is with the Ethyl Corp., New York. He had been associated with the Hodges Research & Development Co.

William H. Bauer is no longer associated with the Treadwell Construction Co., Chicago. He has accepted a position with the Pressed Steel Car Co., Inc., Chicago Steel Tank Div., as sales engineer.

W. Hilgers has been made superintendent for the Otavi Minen & Eisenbahn Gesellschaft., Frankfurt/Main, Germany.

Edward W. Hill has joined the Koppers Co., Inc., Freyn Engineering Dept., Chicago. Mr. Hill had been connected with the Loftus Engineering Corp., Pittsburgh.

Robert N. Langham has been appointed to the staff of the Y-12 plant, an atomic energy installation at Oak Ridge, Tenn.

William R. Smith has joined the General Electric Co., Richland, Wash.

Richard P. Seelig, formerly a vice-president of American Electro Metal Corp., Yonkers, has been named executive vice-president, Chromalloy Corp. Mr. Seelig has been active in the field of powder metallurgy and related fields for the past 15 years.

Howard Storm, formerly assistant general manager, Colorado Iron Works Co., Denver, is now metallurgical engineer with the Atomic Energy Commission, Raw Materials Branch, Washington, D. C.

E. D. Martin is superintendent, research and development dept., Inland Steel Co., Indiana Harbor, Ind.

Owen K. Shupe has accepted the position of metallurgist with the General Electric Co., Richland, Wash.

Richard J. Menze is metallurgist for the Magnet Cove Barium Corp., Malvern, Ark.

M. Merlub-Sobel is on Sabbatical leave from the Hebrew Institute of Technology and will be in America for the school year 1952 to 1953.

Robert J. Morris has resigned as metallurgist with the Allison Div., General Motors, and has accepted a position with the P. R. Mallory Co., Indianapolis.

Rip V. Thompson, formerly mill superintendent for Metal Producers, Millford, Utah, is now metallurgist with Manganese, Inc., Henderson, Nev.

Ferdinand L. Vogel, Jr., formerly instructor of metallurgy at the University of Pennsylvania, is now with the dept. of chemical and metallurgical research, Bell Telephone Laboratories, Murray Hill, N. J.

Stanley W. Porembka has joined the staff of Battelle Memorial Institute, Columbus, Ohio.

John S. McLean is assistant blast furnace manager with the Steel Co. of Wales, Port Talbot.

Frank E. McGinley is now with the AEC at Plant City, Fla. as metallurgical engineer.

E. Raymond Jerome, Jr. is assistant to the general manager, Victor Ring Traveler Co., Providence, R. I.

F. K. Younger has accepted a position as metallurgist with the Federated Metals Div. of American Smelting & Refining Co., Pittsburgh.

James S. Vanick, research metallurgist, the International Nickel Co., N. Y., was the recipient of an annual award given by the Gray Iron Founder's Society for contributions to the gray iron foundry industry. Mr. Vanick is Secretary-Treasurer of the New York Section, AIME.

Andre Guinier, professor of physics, Sorbonne, and professor of X-ray, Conservatoire National des Arts et Metiers, Paris, has been appointed visiting professor of physical metallurgy, in the dept. of mining and metallurgical engineering, University of Illinois.

Thomas Stephan Howald is mechanical metallurgist for Chase Brass & Copper, Inc., Euclid, Ohio.

Oliver B. J. Fraser, assistant manager of development and research div., International Nickel Co., Inc., was the recipient of the Samuel Wylie Miller award of the American Welding Society "for meritorious achievements in the art of welding."

Ralph D. Barer, formerly metallurgist with the Consolidated Mining & Smelting Co., Trail, B. C., is now supervisor of the metals-chemistry section, Pacific Naval Laboratory, Esquimalt, B. C.

George P. Krumlauf is associated with the Republic Steel Corp., Cleveland, as metallurgical engineer in the pig iron div.

Irving A. Dickter has accepted a position as supervisor, flamatic laboratory, Cincinnati Milling & Grinding Machines Co., Cincinnati.

C. F. HOOD

Clifford F. Hood has been named president of the U. S. Steel Corp. and also a member of the board of directors and finance committee. Mr. Hood had been vice-president, operations. In 1950 Mr. Hood was president of the former Carnegie-Illinois Steel Corp. and when that firm became a part of the U. S. Steel Co., he was made executive vice-president, operations.

L. F. Hickernell, chief engineer, Anaconda Wire & Cable Co., Hastings-on-Hudson, N. Y., has been elected director of the board of directors of the AIEE.

Thomas E. Lloyd, formerly assistant to the president, Salem-Brosius, Inc., Pittsburgh, has been named manager of sales.

Arthur Hjortsberg, U. S. Steel Co., has been made assistant chief metallurgical engineer, Pittsburgh. He had been assistant general superintendent at the Gary works.

Amos J. Shaler has been appointed professor of metallurgy and chief of the div. of metallurgy at the Pennsylvania State College. Professor Shaler was recently scientific liaison officer with the ONR in Europe. In 1951 he became associate professor at MIT and served in this capacity until his present appointment.

M. B. Nicholls, president of Nicholls Minerals Corp., Cincinnati, recently went to Lima, Peru in connection with developments of heavy mineral values in that country.

H. Edward Flanders, professor of metallurgy, University of Utah, has won second place prize for a research paper on welding. The International Prize Competition was sponsored by Eutectic Welding Alloys Corp., New York.

G. F. Sipp, associated with Rheem Metalurgica, S. A., Rio de Janeiro, is now located in New York with Rheem International.

Obituaries

W. T. Burns (Member 1903) has died. Born in 1867, Mr. Burns was first employed as a telephone exchange manager by the N. W. Telephone Co. of Minneapolis. In 1890 he joined the M. S. Parker Co. Three years later he became clerk and foreman for the electrolytic copper refinery of the Boston & Montana Co., Great Falls, Mont. He was later made general foreman. In 1916 Mr. Burns joined the Anaconda Copper Mining Co. and ten years later became associated with the Raritan Copper Works, Perth Amboy, N. J.

Hugh J. Fraser (Member 1940) died on Aug. 22, 1952. Mr. Fraser was born at Ontario in 1897 and attended Queen's University. He graduated in 1923 with the degree of B.Sc. After graduation he joined the Huntington works of International Nickel Co. as a metallurgical assistant. From 1924 to 1926 he was in charge of seamless tubing development and was then made combustion engineer. In 1935 Mr. Fraser was transferred to the executive office in New York as assistant manager of production. He was later appointed vice-president.

John Hudson Hall (Member 1929) has died, according to Institute records. Born at Tarrytown-on-Hudson in 1896, Mr. Hall graduated from Harvard in 1919. Mr. Hall was assistant secretary, United Verde Copper Co., New York and assistant manager, domestic copper sales for the same firm.

W. L. Odom (Member 1951), chief, development and research, Gary works, U. S. Steel Co., died in August 1952. He was a graduate of Purdue University and then joined the Gary works, as metallurgical observer. He was made chief observer and then metallurgical laboratory foreman. In 1944 Mr. Odom was transferred to the Geneva Steel Co. as chief metallurgist. A short time later he returned to Gary as assistant to chief metallurgist and in 1948 was named chief control metallurgist.

NECROLOGY

Date Elected	Name	Date of Death
1949	George M. Brown	Unknown
1907	John A. Church	Nov. 10, 1952
1901	Lucien Eaton, Sr.	Dec. 9, 1952
1919	George M. Enos	Oct. 27, 1952
1947	Robert Gale, Jr.	Sept. 6, 1952
1921	S. B. Goldberg	Apr. 11, 1952
1943	Jerome B. Grigg	Oct. 4, 1952
1943	Julius A. Heeren	Unknown
1944	Harold E. Hostetter	Nov. 11, 1952
1917	L. G. Johnson	Nov. 16, 1952
1936	McHenry Mosier	Oct. 19, 1952
1923	A. D. Riley	Oct. 25, 1952
1937	Otto D. Rohlfs	Sept. 25, 1952
1939	T. K. Stinson	Oct. 14, 1952
1948	Howard L. Sweet	September 1952
1952	V. L. Vasilievich	Nov. 11, 1952
1914	Joel H. Watkins	Oct. 27, 1952
1939	Job H. Winwood	Nov. 4, 1952

Proposed for Membership — Metals Branch AIME —

Total AIME membership on Oct. 31, 1952 was 18,333; in addition 1697 Student Associates were enrolled.

ADMISSIONS COMMITTEE

T. D. Jones, Chairman; Thomas G. Moore, Vice-Chairman; Harold S. Bell, F. W. Hanson, R. H. Chadwick, T. W. Nelson, J. H. Scaff, John T. Sherman, A. C. Brinker, Ivan Given, C. A. R. Lambly, G. P. Lutjen, E. A. Prentis, and C. Leslie Rice, Jr.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

Alabama
Auburn—Sturm, Rolland G. (M)

California
Berkeley—Beaver, George L. (A)
Ontario—Lohrey, Clarence R. (M)
Van Nuys—Walden, Edward (M)
Whittier—Paine, Roy E. (M) (R. C/S—S-M)

Illinois
Chicago—Kohan, Leonard R. (J)
Chicago—Weigert, Karl M. (M)
Lansing—Franzen, Erwin F. (A)

Indiana
East Chicago—Bare, Earle H., Jr. (J)

Michigan
Royal Oak—Hunnicutt, Richard P. (J) (C/S—S-J)

Montana
Great Falls—DeArmond, Charles F. (M)

New Jersey
Hawthorne—Isleib, Charles R. (J)
Matawan—Rodman, Harry G. (J)
Metuchen—Hoehne, Claren H. (J) (C/S—S-J)
Trenton—Cole, William C. (J)

New York
Alfred—Soxman, Edwin J. (J)
Brooklyn—Castiglioni, Salvatore J. (J) (R. C/S—S-J)
Brooklyn—Galvez, Matthew J. (J)
Brooklyn—Sutinen, Clemens C. (J) (C/S—S-J)
Endicott—Meyer, James S. (M)
Youngstown—Reynolds, Samuel H. (M)

North Carolina
Winston-Salem—Davis, Moss V. (A)

Ohio
Columbus—Porembka, Stanley W., Jr. (J) (C/S—S-J)
Piqua—Achbach, William P. (J)
Shaker Heights—Scott, Theodore G., Jr. (M) (R. C/S—J-M)

Pennsylvania
Boyertown—Kawecki, Henry C. (M)
Coatesville—Bigler, Clarke A. (J)
Fairless Hills—Loughrey, Robert P. (J)
Hatboro—Volk, Donald J. (A)
Latrobe—Sherman, Charles W. (R. C/S—S-M)
Merion—Hanak, Richard J. (M)

Texas
El Paso—Heller, Carl W. (M)
El Paso—Lucker, Gerald D. (M)
El Paso—Nobles, Jesse M. (A)
El Paso—Prescott, Harry T. (M) (R. C/S—J-M)

Utah
Salt Lake City—Hilton, Charles C. (M)

Virginia
Falls Church—Wardell, Richard N. (J) (C/S—S-J)

Wisconsin
Milwaukee—Michael, Arthur B. (J) (R. C/S—S-J)

Canada
Ontario, Welland—Kellam, Franklin W. (M)

England
Kent—Almond, John K. (J)

Germany
Nurnberg—Brenthol, Franz C. (M)

Coming Events

Jan. 6, 1953, Society for Applied Spectroscopy, Socony-Vacuum Training Center, New York.

Jan. 7, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Jan. 14, AIME, Connecticut Section, Hammond Metallurgical Laboratory, Yale University, New Haven, Conn.

Jan. 14, AIME, Bessemer Committee, Duquesne Club, Pittsburgh.

Jan. 14-16, American Society of Photogrammetry, Shoreham Hotel, Washington, D. C.

Jan. 19-22, Plant Maintenance Show, Public Auditorium, Cleveland.

Jan. 21, AIME, National Open Hearth Steel Committee, Roger Young YMCA, Los Angeles.

Jan. 21-23, Society of Plastics Engineers, Inc., annual technical conference, Hotel Statler, Boston.

Jan. 26-30, American Society of Heating and Ventilating Engineers, international heating and ventilation exposition, International Amphitheatre, Chicago.

Feb. 4, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Feb. 16-19, AIME, annual meeting, Statler Hotel, Los Angeles.

Mar. 4, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Mar. 8-11, American Institute of Chemical Engineers, Buena Vista Hotel, Biloxi, Miss.

Mar. 11, AIME, Connecticut Section, Waterbury, Conn.

Mar. 16-20, National Assn. of Corrosion Engineers, annual conference and exhibition, Hotel Sherman, Chicago.

Mar. 18, AIME, National Open Hearth Steel Committee, Roger Young YMCA, Los Angeles.

Mar. 23-27, ASM, western metal congress and exposition, Pan-Pacific Auditorium, Los Angeles.

April-May, Empire Mining and Metallurgical Congress, Australia and New Zealand.

Apr. 1, AIME, Chicago Section, Chicago Bar Assn., Chicago.

Apr. 12-May 23, Empire Mining and Metallurgical Congress, Australia-New Zealand.

Apr. 20-22, AIME, National Open Hearth and Blast Furnace, Coke Oven and Raw Materials Conference, Hotel Statler, Buffalo.

Apr. 25-May 10, Liege International Fair, Liege, Belgium.

Apr. 26-29, American Institute of Chemical Engineers and **Chemical Institute of Canada,** Royal York Hotel, Toronto.

May 14-16, AIME, Pacific Northwest Metals and Mineral Conference, joint meeting, Metals Branch and Industrial Minerals Div., Ben Franklin Hotel, Seattle.

May 18-22, National Materials Handling Exposition, Convention Hall, Philadelphia.

May 20, AIME, National Open Hearth Steel Committee, field trip.

June 3-7, Chemical Institute of Canada, Windsor, Ont.

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